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STATIONARY SOURCE SAMPLING REPORT

EEI REF. NO. 5448

GENERAL REFINERY PLANT
GARDEN CITY, GEORGIA

BENZENE, MERCURY, TOLUENE, TRIETHYLAMINE,
AND XYLENE EMISSIONS TESTING

CONDENSER EXHAUST AND OIL POLISHER OUTLET

Performed For: RESOURCES CONSERVATION CO.

FEBRUARY 26-28, 1987

RECEIVED

FEB 15 1988

CHEMICAL AND METAL
CONTROL UNIT

REPORT CERTIFICATION

The sampling and analysis performed for this report was carried out under my direction and supervision.

Date March 31, 1987

Signature B. Dwain Ritchie

B. Dwain Ritchie

I have reviewed all testing details and results in this test report and hereby certify that the test report is authentic and accurate.

Date March 31, 1987

Signature Walter S. Smith

Walter S. Smith, P.E.

TABLE OF CONTENTS

	<u>PAGE</u>
INTRODUCTION	
1.1 Background	1-1
1.2 Process Test Locations	1-1
1.2.1 Condenser Exhaust	1-1
1.2.2 Oil Polisher Outlet	1-1
1.3 Test Participants	1-1
SUMMARY OF RESULTS	2-1
SAMPLING AND ANALYTICAL PROCEDURES	
3.1 Sampling Equipment	3-1
3.2 Volumetric Gas Flow Rates	3-1
3.2.1 Condenser Exhaust	3-1
3.2.2 Oil Polisher Outlet	3-1
3.3 Sample Acquisition	3-2
3.3.1 Condenser Exhaust	3-2
3.3.2 Oil Polisher Outlet	3-3
3.4 Sample Recovery	3-4
3.5 Sample Analysis	3-4
APPENDICES	
A. Test Results	
1. Condenser Exhaust	
2. Oil Polisher Outlet	
B. Field Data	
1. Condenser Exhaust	
2. Oil Polisher Outlet	
C. Analytical Data	
D. Calibration Data	
E. Sampling and Analytical Procedures	

INTRODUCTION

1.1 Background. Resources Conservation Co. was contracted by the United States Environmental Protection Agency to conduct cleanup operations at the defunct General Refinery Plant in Garden City, Georgia. Cleanup operations include the treatment and removal of industrial waste abandoned on the plant site.

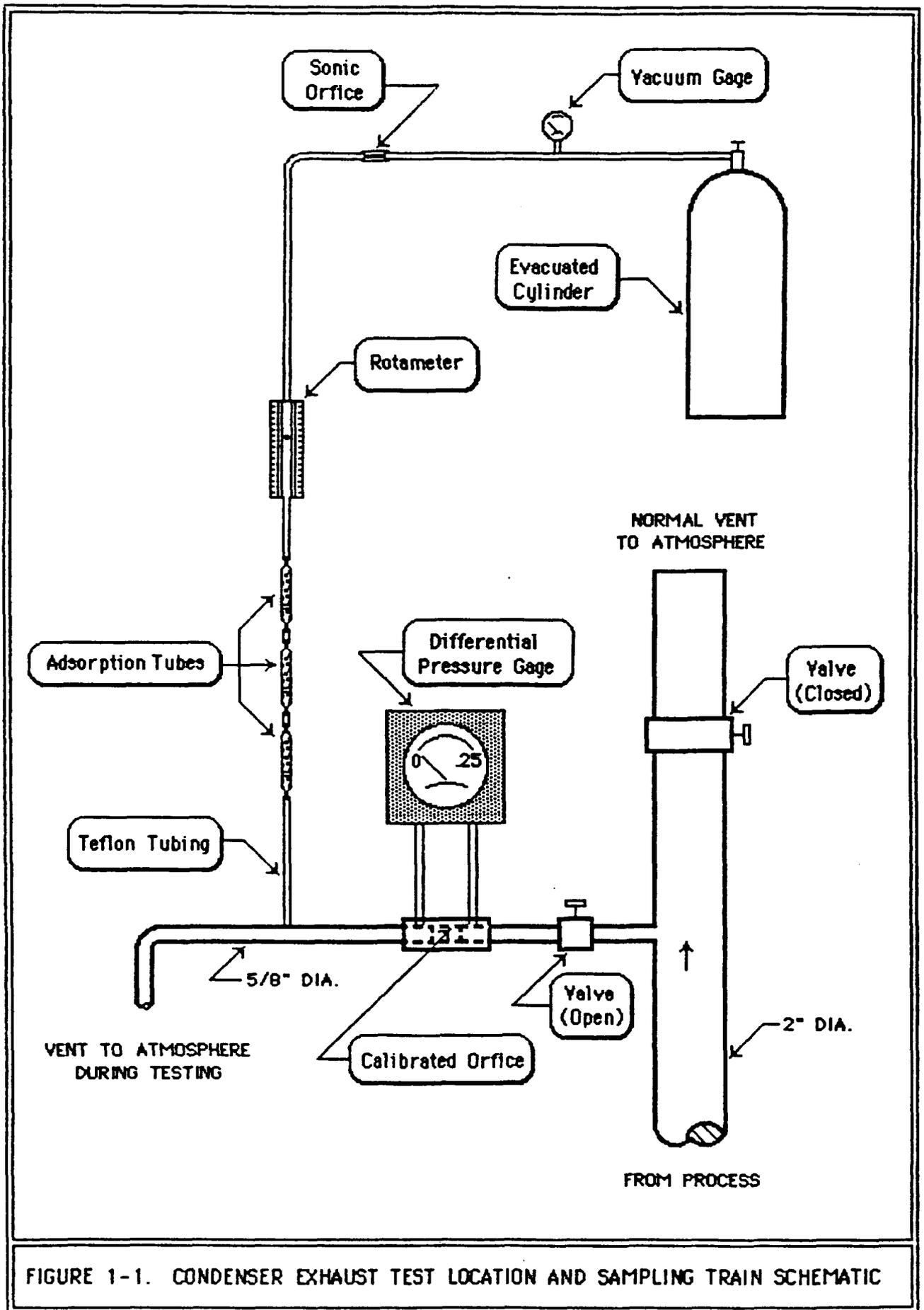
To prepare a sludge waste product for removal from the site, the oil, moisture, and solids components are separated using a series of solvent, heating, and condensing processes. Resource Conservation Co. employed Entropy Inc. to measure emissions of benzene, mercury, toluene, triethylamine, and xylene at two of the process exhausts.

1.2 Process Test Locations

1.2.1 Condenser Exhaust. The combined effluent from several condensers was diverted from the normal exhaust path through a tee extension, as shown in Figure 1-1. The test equipment installed in the tee extension allowed the concurrent measurement of velocity and collection of emission samples. During each of four tests, three different pollutant-specific sampling trains were operated sequentially. Testing at the condenser exhaust was conducted on February 26, 27, and 28, 1987.

1.2.2 Oil Polisher Outlet. The oil polisher effluent, primarily steam, was sampled on February 27, 1987, during one test which was made possible by a process alteration arranged by Resource Conservation Co. Equipment inserted in the outlet pipe through two access ports allowed the concurrent collection of samples and measurement of velocity. The sampling arrangement is depicted in Figure 1-2.

1.3 Test Participants. Table 1-1 lists the personnel present during the test program.



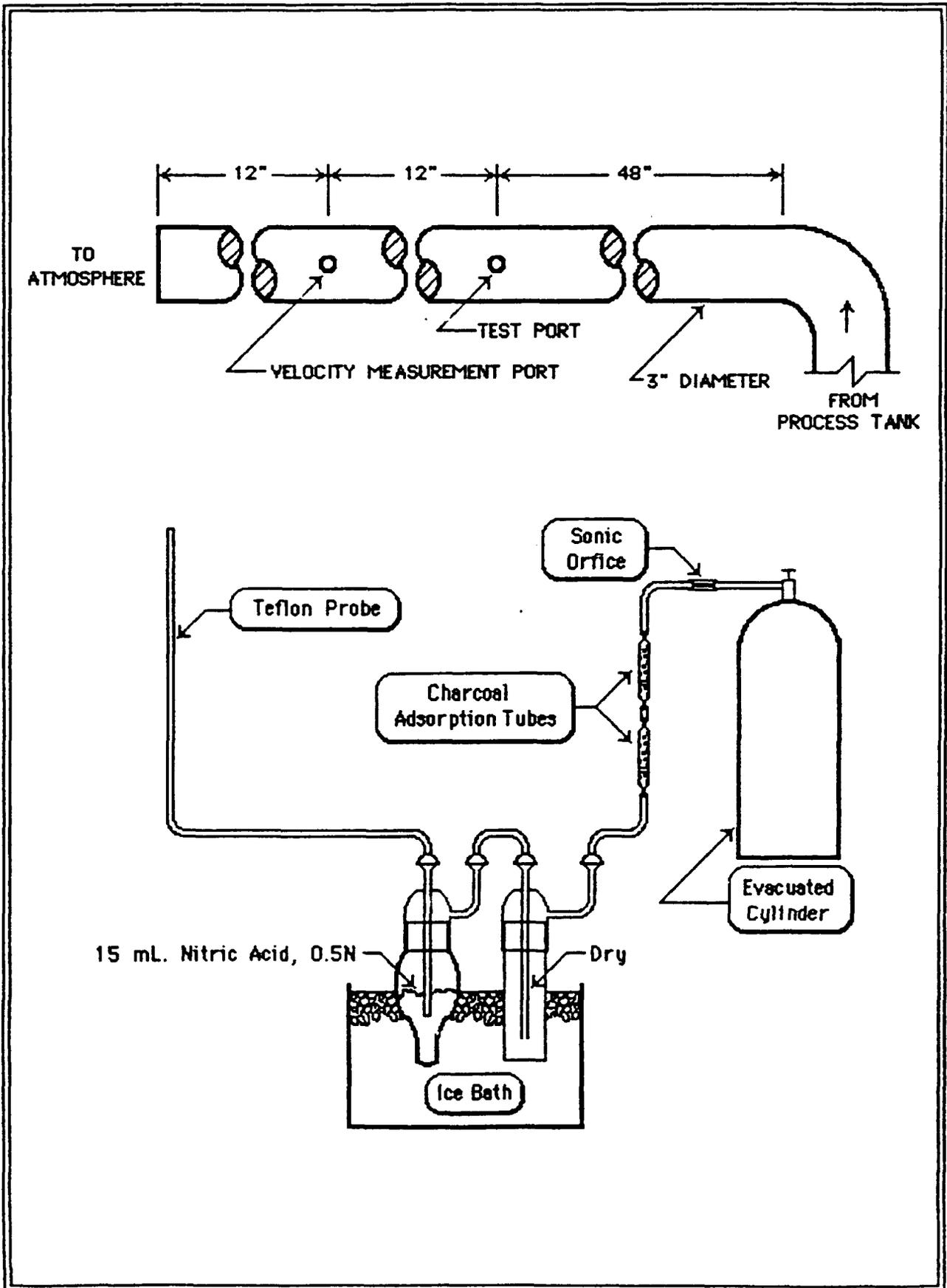


FIGURE 1-2. OIL POLISHER OUTLET TEST LOCATION AND SAMPLING TRAIN SCHEMATIC

TABLE 1-1
TEST PARTICIPANTS

Resources Conservation Co.

Test Coordinators

Mark Tose
R. Reams Goodloe, Jr.

Entropy Inc.

Project Supervisor

B. Dwain Ritchie

Sampling Team Leaders

Anthony L. Mastrianni
Brent W. Hall
Robert W. Metz

SUMMARY OF RESULTS

Table 2-1 summarizes the results of emission measurements performed at the condenser exhaust and the oil polisher outlet on February 27, 1987. Detailed test results are tabulated in Appendix A; field and analytical data are provided in Appendix B.

TABLE 2-1
SUMMARY OF EMISSIONS

	Run				Average
	1	2	3	4	
<u>Condenser Exhaust</u>					
Concentration, ppmvd					
Benzene	321	321	339	311	323
Mercury	< 0.00496	< 0.00496	< 0.00496	< 0.00496	< 0.00496
Toluene	164	144	145	132	146
Triethylamine	22,560	13,235	29,928	29,003	23,682
Xylene	200	182	191	161	184
Emission Rate, Lb/Hr					
Benzene	0.00127	0.00127	0.000926	0.00108	0.00114
Mercury	< 0.00000052	< 0.00000041	< 0.00000042	< 0.00000037	< 0.00000043
Toluene	0.000769	0.000676	0.000469	0.000540	0.000614
Triethylamine	0.0985	0.0492	0.131	0.103	0.0954
Xylene	0.00108	0.000983	0.000710	0.000763	0.000884
<u>Oil Polisher Outlet</u>					
Concentration, ppmvd					
Benzene	39.3				
Mercury	< 0.0677				
Toluene	1,502				
Triethylamine	20,130				
Xylene	8,271				
Emission Rate, Lb/Hr					
Benzene	0.000473				
Mercury	< 0.00000210				
Toluene	0.0214				
Triethylamine	0.314				
Xylene	0.135				

SAMPLING AND ANALYTICAL PROCEDURES

3.1 Sampling Equipment. The arrangements of sampling equipment at the condenser exhaust and the oil polisher outlet were shown in Figures 1-1 and 1-2. All equipment was manufactured by Nutech Corp. or Entropy Inc. Pertinent calibration data are presented in Appendix D.

3.2 Volumetric Gas Flow Rates.

3.2.1 Condenser Exhaust. As illustrated in Figure 1-1, gas flow rate was measured using a calibrated orifice; orifice pressure drop readings were recorded at two-minute intervals throughout each test run. The molecular weight of the sample gas was calculated using the molecular weights and concentrations of the significant constituents of the gas (ambient air and triethylamine). The gas moisture content was calculated assuming saturation. The volumetric gas flow rate was determined using these values in the following equation:

Equation 1

$$Q = K_m \sqrt{\frac{T_{abs} * \text{delta } p}{P_{abs} * \text{Mol. Wt.}}}$$

Where:

Q	= flow rate, cfm
K_m	= orifice calibration coefficient
T_{abs}	= absolute temperature, °R
P_{abs}	= absolute pressure, inches Hg
delta p	= orifice pressure drop, inches H ₂ O
Mol. Wt.	= molecular weight of gas, lb/lb mole, wet

3.2.2 Oil Polisher Outlet. Gas flow rate was measured by taking readings at one-minute intervals throughout the single test run using an S-type pitot tube inserted into the oil polisher outlet duct through a drilled port 12 inches downstream of the sampling port (see Figure 1-2). The molecular weight of the sample gas was that of ambient air. The gas moisture content was calculated assuming saturation. The following equations were used to calculate the volumetric gas flow rate:

$$\text{Equation 2} \quad v_s = 85.49 * C_p * \frac{\text{avg. delta } p * T_{\text{abs}}}{P_{\text{abs}} * \text{Mol. Wt.}}$$

Where: v_s = average velocity, fps
 85.49 = pitot calibration coefficient
 avg. delta p = differential pressure, inches H_2O
 T_{abs} = absolute temperature, $^{\circ}R$
 P_{abs} = absolute pressure, inches Hg
 Mol. Wt. = molecular weight of gas, lb/lb mole, wet

$$\text{Equation 3} \quad Q_{sd} = \frac{60}{144} * M_{fd} * v_s * A * \frac{528}{T_s} * \frac{P_s}{29.92}$$

Where: Q_{sd} = dry volumetric flow rate at standard conditions, cfm
 $60/144$ = seconds per minute/cubic inches per cubic foot
 M_{fd} = dry mole fraction of the flue gas
 v_s = average gas velocity, fps
 A = duct cross sectional area, square feet
 528 = standard absolute temperature, $^{\circ}R$
 29.92 = standard absolute pressure, inches Hg

3.3 Sample Acquisition.

3.3.1 Condenser Exhaust. The condenser exhaust gas was sampled using the arrangement shown in Figure 1-1. The sample gas was diverted from the normal exhaust path by closing the exhaust valve and opening the ball valve in the tee extension in which the sample line was installed. Extracted by the negative pressure of an evacuated cylinder, the sample gas flowed through three adsorption tubes in series, a rotameter, a sonic orifice, a vacuum gauge, and into the cylinder.

Three pollutant-specific sampling trains were operated sequentially to collect the samples. Although duplicate samples (sample lines A and B) were taken during the operation of each train, only the samples from line A were analyzed; the duplicates were retained as backups. Table 3-1 summarizes the arrangements of the various adsorbent media for each sampling train (also documented on field data records, Appendix B) and states the capture objectives of each medium.

TABLE 3-1
CONDENSER EXHAUST ADSORBENT SAMPLING MEDIA AND CAPTURE OBJECTIVES

	<u>Sample Collection Medium</u>	<u>Capture Objective</u>
Train A		toluene, benzene, xylene
Position 1	charcoal	
Position 2	charcoal	
Position 3	charcoal	
Train B		triethylamine
Position 1	silica gel	
Position 2	silica gel	
Position 3	silica gel	
Train C		mercury
Position 1	silvered Chromosorb P	
Position 2	silvered Chromosorb P	
Position 3	silica gel	

3.3.2 Oil Polisher Outlet. The oil polisher outlet gas was sampled by the insertion of a sampling probe into the test port, as shown in Figure 1-2. Extracted by the negative pressure of an evacuated cylinder, the sample gas passed through a Mae West impinger charged with 15 mL of 0.5N nitric acid and a dry impinger (both submerged in ice), two adsorption tubes charged with activated charcoal, a sonic orifice, and into the cylinder. Table 3-2 identifies the capture objectives for each of the sampling train components and capture media.

TABLE 3-2
OIL POLISHER OUTLET SAMPLING TRAIN COMPONENTS AND CAPTURE OBJECTIVES

<u>Train Component</u>	<u>Sample Collection Medium</u>	<u>Capture Objective</u>
Mae West impinger	0.5N Nitric Acid Reagent	benzene toluene xylene triethylamine gaseous mercury
adsorbent tubes	charcoal	benzene toluene xylene

3.4 Sample Recovery. The samples were transferred from the test site to the laboratory on ice in a cooler. The adsorbent tubes were transferred to a freezer for storage. The impinger reagent from the oil polisher outlet (OP) test was stored in a refrigerator after recovery. The reagent from the OP sample was bilayered with 47 mL in the aqueous layer and 3 mL in the organic phase. Blue ice packs were used to keep the samples cool during shipment to Oxford Laboratories. After receipt at Oxford Laboratories, the samples were stored in a freezer or refrigerator until analysis. Chain-of-custody records for the transfer of the samples to Oxford Laboratories are presented in Appendix C.

3.5 Sample Analysis. The silica gel adsorbent tubes and the aqueous portion of the OP reagent were analyzed for triethylamine according to the protocol in NIOSH Method 221. The silvered Chomosorb P tubes were desorbed with acid and analyzed for mercury by cold vapor atomic absorption. The aqueous portion of the OP reagent was also analyzed by cold vapor atomic absorption for mercury. Charcoal adsorbent tubes were analyzed for benzene, toluene, and xylenes by the protocol of NIOSH Method 1501. An aliquot of the OP reagent organic phase was diluted in methanol. A 50 uL aliquot of the methanol sample was placed in distilled water and analyzed by purge trap GC/PID. For all the adsorbent train analyses, the second "b" tube was analyzed separately from the primary "a" tube. The analytes were not detected on any of the "b" tubes, thus demonstrating that the pollutants were effectively captured on the "a" tubes.

APPENDIX A.1

A. TEST RESULTS

1. Condenser Exhaust

GAS FLOW RATE TABULATIONS

Condenser Exhaust

Water %: 1.1

Mfd = 0.989

Sample	Orifice Delta P	Gas Temp F	Baro. Press. in. Hg	Static Press. in. H2O	Molecular Weight lb/lb-mole		- Air Flows -	
					Dry	Wet	ACFM Wet	SCFM Dry
CE-1A	0.307	50	30.4	-4.2	30.50	30.36	0.317	0.327
CE-1B	0.220	50	30.4	-4.0			0.268	0.277
CE-1C	0.330	50	30.4	-4.3			0.329	0.339
CE-2A	0.301	50	30.4	-4.1	29.78	29.65	0.318	0.327
CE-2B	0.157	50	30.4	-4.3			0.230	0.236
CE-2C	0.196	50	30.4	-4.2			0.257	0.264
CE-3A	0.148	50	30.4	-3.0	31.01	30.87	0.218	0.225
CE-3B	0.226	50	30.4	-5.0			0.270	0.278
CE-3C	0.211	50	30.4	-4.4			0.261	0.269
CE-4A	0.237	46	30.4	-4.2	30.94	30.80	0.276	0.286
CE-4B	0.147	48	30.4	-4.5			0.218	0.225
CE-4C	0.164	46	30.4	-4.6			0.229	0.238

FIELD DATA & RESULTS TABULATION
 General Refinery Plant
 Garden City, Georgia

<u>Run</u>	<u>Sampling Location</u>	<u>Operator</u>
CE-1Aa	Condenser Exhaust	B. Dwain Ritchie
CE-2Aa	Condenser Exhaust	Brent W. Hall
CE-3Aa	Condenser Exhaust	Brent W. Hall
CE-4Aa	Condenser Exhaust	Brent W. Hall

	<u>CE-1Aa</u>	<u>CE-2Aa</u>	<u>CE-3Aa</u>	<u>CE-4Aa</u>
Run Date	2/26/87	2/26/87	2/27/87	2/28/87
Run Start Time	1614	2233	315	1115
Run Finish Time	1644	2303	345	1145
Theta Net Run Time, Minutes	30	30	30	30
Qsd Flue Gas Flow Rate, Dry SCFM*	0.327	0.327	0.225	0.286
Vc Avg. Calibration Vol., Milliliters	39	39	39	39
Y Calibration Meter Factor, Gamma	1.034	1.034	1.034	1.034
Vsc Volume Of Gas Sampled, DSL*	1.21	1.21	1.21	1.21
<u>Benzene</u>				
ForWt Formula Weight, Lb/Lb-Mole	78.0	78.0	78.00	78.00
mg Catch, Milligrams	1.26	1.26	1.33	1.22
ppmvd Concentration, ppmv, Dry	321	321	339	311
Lb/Hr Emission Rate, Lb/Hour	0.00127	0.00127	0.000926	0.00108
<u>Toluene</u>				
Formula Weight, Lb/Lb-Mole	92.13	92.13	92.13	92.13
Catch, Milligrams	0.76	0.668	0.673	0.610
Concentration, ppmv, Dry	164	144	145	132
Emission Rate, Lb/Hour	0.000769	0.000676	0.000469	0.000540
<u>Xylene</u>				
Formula Weight, Lb/Lb-Mole	106.16	106.16	106.16	106.16
Catch, Milligrams	1.07	0.971	1.02	0.862
Concentration, ppmv, Dry	200	182	191	161
Emission Rate, Lb/Hour	0.00108	0.000983	0.000710	0.000763

* 68 Deg. F - 29.92 Inches of Mercury (Hg)

FIELD DATA & RESULTS TABULATION
 General Refinery Plant
 Garden City, Georgia

<u>Run</u>	<u>Sampling Location</u>	<u>Operator</u>
CE-1Ba	Condenser Exhaust	B. Dwain Ritchie
CE-2Ba	Condenser Exhaust	B. Dwain Ritchie
CE-3Ba	Condenser Exhaust	Brent W. Hall
CE-4Ba	Condenser Exhaust	Brent W. Hall

		<u>CE-1Ba</u>	<u>CE-2Ba</u>	<u>CE-3Ba</u>	<u>CE-4Ba</u>
	Run Date	2/26/87	2/26/87	2/27/87	2/28/87
	Run Start Time	1717	2328	420	1200
	Run Finish Time	1733	2348	450	1230
Theta	Net Run Time, Minutes	20	20	30	30
Qsd	Flue Gas Flow Rate, Dry SCFM*	0.277	0.236	0.278	0.225
Vc	Avg. Calibration Vol., Milliliters	39	39	39	39
Y	Calibration Meter Factor, Gamma	1.034	1.034	1.034	1.034
Vsc	Volume Of Gas Sampled, DSL*	0.825	0.817	1.25	1.25
	<u>Triethylamine</u>				
ForWt	Formula Weight, Lb/Lb-Mole	101.19	101.19	101.19	101.19
mg	Catch, Milligrams	78.3	45.5	157	152
ppmvd	Concentration, ppmv, Dry	22,560	13,235	29,928	29,003
Lb/Hr	Emission Rate, Lb/Hour	0.0985	0.0492	0.131	0.103

* 68 Deg. F - 29.92 Inches of Mercury (Hg)

FIELD DATA & RESULTS TABULATION

General Refinery Plant

Garden City, Georgia

<u>Run</u>	<u>Sampling Location</u>	<u>Operator</u>
CE-1Ca	Condenser Exhaust	B. Dwain Ritchie
CE-2Ca	Condenser Exhaust	B. Dwain Ritchie
CE-3Ca	Condenser Exhaust	Brent W. Hall
CE-4Ca	Condenser Exhaust	Brent W. Hall

		<u>CE-1Ca</u>	<u>CE-2Ca</u>	<u>CE-3Ca</u>	<u>CE-4Ca</u>
	Run Date	2/26/87	2/27/87	2/27/87	2/28/87
	Run Start Time	1815	19	520	1250
	Run Finish Time	1845	49	550	1320
Theta	Net Run Time, Minutes	30	30	30	30
Qsd	Flue Gas Flow Rate, Dry SCFM*	0.339	0.264	0.269	0.238
Vc	Avg. Calibration Vol., Milliliters	39	39	39	39
Y	Calibration Meter Factor, Gamma	1.034	1.034	1.034	1.034
Vsc	Volume Of Gas Sampled, DSL*	1.21	1.21	1.21	1.21
	<u>Mercury</u>				
ForWt	Formula Weight, Lb/Lb-Mole	200.59	200.59	200.59	200.59
mg	Catch, Milligrams	< 0.00005	< 0.00005	< 0.00005	< 0.00005
ppmvd	Concentration, ppmv, Dry	< 0.00496	< 0.00496	< 0.00496	< 0.00496
Lb/Hr	Emission Rate, Lb/Hour	< 0.000000052	< 0.000000041	< 0.000000042	< 0.000000037

* 68 Deg. F - 29.92 Inches of Mercury (Hg)

APPENDIX A.2

A. TEST RESULTS

2. Oil Polisher Outlet

FIELD DATA & RESULTS TABULATION

PLANT: General Refinery Plant, Garden City, Georgia

RUN	DATE	SAMPLING LOCATION	OPERATOR
OP-1	2/27/87	Oil Polisher Outlet	Robert W. Metz
			OP-1

	Run Start Time		1114
	Run Finish Time		1117
Theta	Net Run Time, Minutes		2.5
Cp	Pitot Tube Coefficient		0.84
Pbar	Barometric Pressure, Inches Hg		30.4
	<u>Tank Sampling Parameters</u>		
V(tank)	Volume of Tank, liters		7.071
t(pre)	Pretest Temperature, deg. F		48
Pa(pre)	Pretest Absolute Pressure, mmHg		772.2
t(post)	Posttest Temperature, deg. F		48
Pa(post)	Posttest Absolute Pressure, mmHg		772.2
Vstd	Volume Sampled, Dry SCF*		0.0294
Vlc	Volume of Water Collected in Impingers, mL		32.0
V	Volume of Water Vapor, SCF*		1.506
%H2O	Moisture Content, Percent By Volume		98.1
Mfd	Dry Mole Fraction		0.019
Md	Dry Molecular Weight, Lb/Lb-Mole		28.84
Ms	Wet Molecular Weight, Lb/Lb-Mole		18.21
Pg	Flue Gas Static Pressure, Inches H2O		0.0
Ps	Absolute Flue Gas Pressure, Inches Hg		30.4
ts	Flue Gas Temperature, Degrees F		212
Delta p	Average Velocity Head, Inches H2O		0.0786
vs	Flue Gas Velocity, Feet/Second		22.18
A	Stack/Duct Area, Square Inches		7.07
Qsd	Volumetric Air Flow Rate, Dry SCFM*		0.991
Qaw	Volumetric Air Flow Rate, Wet ACFM		65.34
	<u>Benzene</u>		
ForWt	Formula Weight, Lb/Lb Mole		78.0
mg	Catch, Milligrams		0.106
ppmvd	Concentration, ppmvd		39.3
Lb/Hr	Emission Rate, Lb/Hr		0.000473
	<u>Mercury</u>		
	Formula Weight, Lb/Lb Mole		200.59
	Catch, Milligrams		< 0.00047
	Concentration, ppmvd		< 0.0677
	Emission Rate, Lb/Hr		< 0.00000210
	<u>Toluene</u>		
	Formula Weight, Lb/Lb Mole		92.13
	Catch, Milligrams		4.791
	Concentration, ppmvd		1.502
	Emission Rate, Lb/Hr		0.0214

* 68 Degrees F -- 29.92 Inches of Mercury (Hg)

OP-1

Triethylamine

Formula Weight, Lb/Lb Mole	101.19
Catch, Milligrams	70.5
Concentration, ppmvd	20.130
Emission Rate, Lb/Hr	0.314

Xylenes

Formula Weight, Lb/Lb Mole	106.16
Catch, Milligrams	30.389
Concentration, ppmvd	8.271
Emission Rate, Lb/Hr	0.135

* 68 Degrees F -- 29.92 Inches of Mercury (Hg)

APPENDIX B.1

B. FIELD DATA

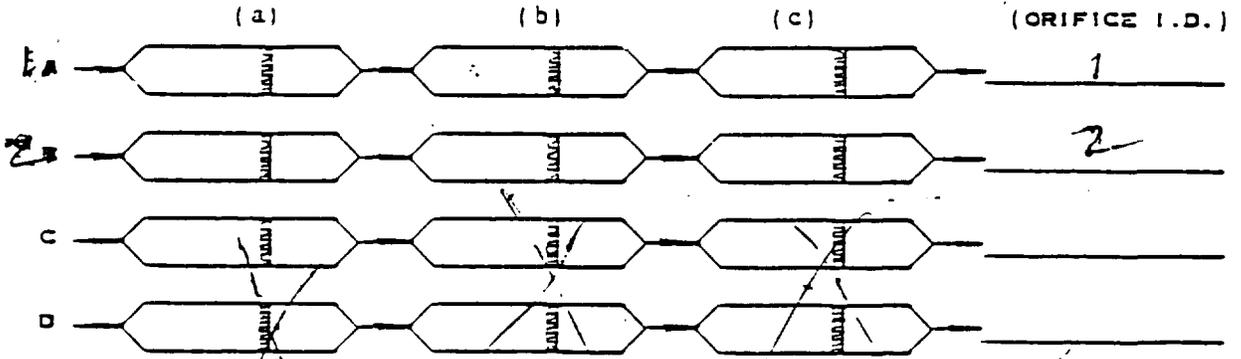
1. Condenser Exhaust

ADSORBENT SAMPLING TRAIN FIELD DATA

CE-1A

COMPANY NAME RESOURCE CONSERVATION
 ADDRESS GADSDEN CITY, GA.
 SAMPLING LOCATION CONDENSED EXHAUST
 DATE 2-26-87 TEAM LEADER TRK TECHNICIANS TM
 BAROMETRIC PRESSURE, IN. HG. (P_{BAR}) 30.4 PUMP # TANK

RUN CE-1A
 TIME START 1614
 TIME FIN. 1644



TYPICAL LABELLING FOR TUBE A (a): 1-A-a

CLOCK TIME	ROTAMETER READINGS			
	1 - SAMPLES	2 - SAMPLES	C - SAMPLES	O - SAMPLES
3:00	47 (C/M/A)	34		
5	42	34		
10	42	43		
15	42	45		
20	44	43		
25	44	42		
30 OFF				

30
(MIN) NET TIME, MINUTES

CALI- BRATION	AMB. TEMP. (Ca)	-A-		-B-		-C-		-O-	
		TIME SECONDS (SEC)	VOLUME MILS (Vc)	TIME SECONDS	VOLUME MILS	TIME SECONDS	VOLUME MILS	TIME SECONDS	VOLUME MILS
INITIAL	52°F	BYRON	38	NA	38				
FINAL	47	MODEL 90	40		38				
AVERAGE	50	60	39	60	38				
VOLUME LITERS (Va)			1		1		1		1
VOLUME STANDARD (V _{sc})			dsl		dsl		dsl		dsl

$$V_a = \text{MIN.} \cdot \frac{50}{1000} \cdot V_c / \text{SEC}$$

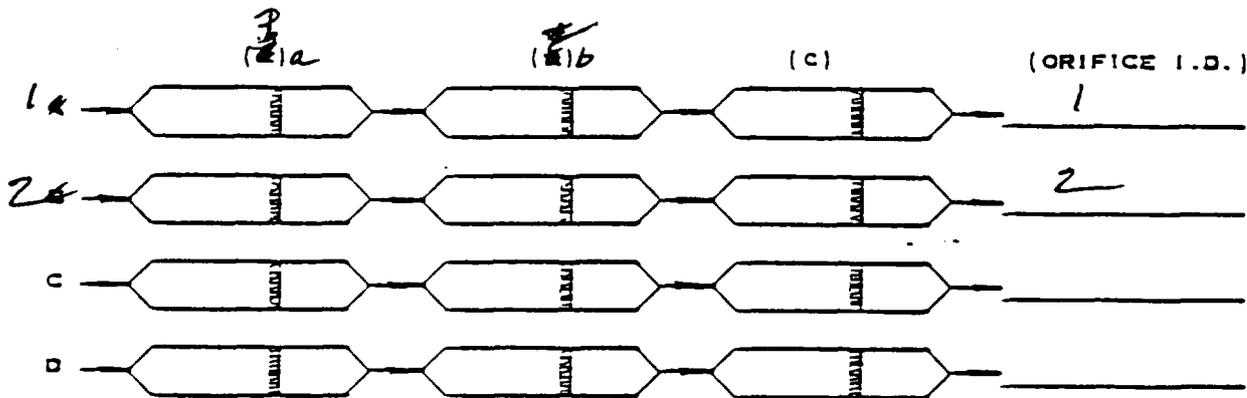
$$V_{sc} = 17.64 \cdot \frac{P_{bar}}{(C_a + 460)} \cdot V_a$$

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ADSORBENT SAMPLING TRAIN FIELD DATA

COMPANY NAME <u>RESOURCE CONSERVATION</u>		RUN <u>CE-1B</u>
ADDRESS <u>GARDEN CITY, GA.</u>		TIME START <u>1713</u>
SAMPLING LOCATION <u>CONDENSER EXHAUST</u>		TIME FIN. <u>1733</u>
DATE <u>2-26-87</u>	TEAM LEADER <u>DK</u>	TECHNICIANS <u>TM</u>
BAROMETRIC PRESSURE, IN. HG. (P _{BAR}) <u>30.4</u>		PUMP # <u>M-25 TRAK</u>



TYPICAL LABELLING FOR TUBE A (a): 1-A-a

CLOCK TIME HOURS	ROTAMETER READINGS			
	1- SAMPLES	2- SAMPLES	C- SAMPLES	D- SAMPLES
0	46	46		
5	46	46		
10	46	46		
15	46	46		
20/DEF				

20
NET TIME, MINUTES
(MIN)

Make 90

CALI- BRATION	AMB. TEMP. (C _a)	1		2		C	D
		TIME SECONDS (SEC)	VOLUME MILS (V _C)	TIME SECONDS (SEC)	VOLUME MILS (V _C)		
INITIAL	46		40 4/min	39 4/min			
FINAL	44		38	39			
AVERAGE	45	60	39	60	39		
VOLUME LITERS (V _a)			1	1	1	1	
VOLUME STANDARD (V _{SC})			dsl	dsl	dsl	dsl	

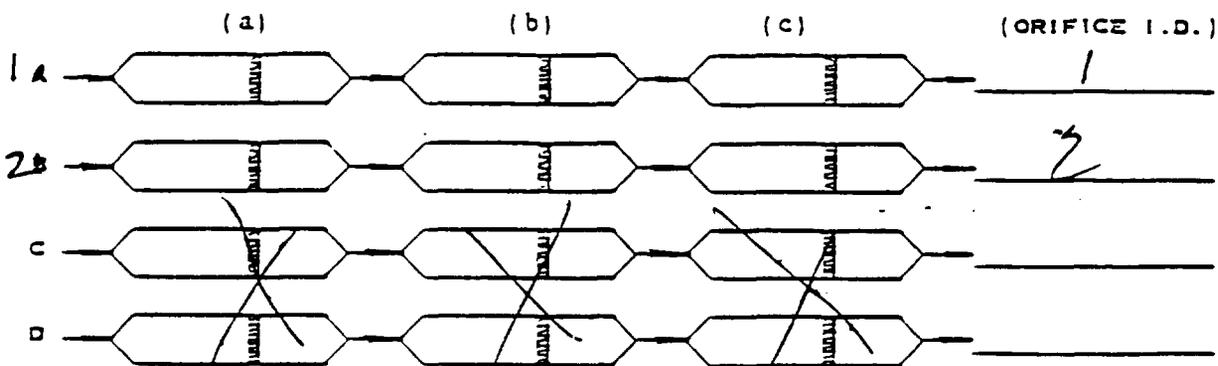
$$V_a = \text{MIN.} \cdot \frac{60}{1000} \cdot V_c / \text{SEC}$$

$$V_{SC} = 17.64 \cdot \frac{P_{bar}}{(C_a - 460)} \cdot V_a$$

ENTROPY

ADSORBENT SAMPLING TRAIN FIELD DATA

COMPANY NAME: RESOURCE CONSERVATION RUN CE-1C
 ADDRESS: GARDEN CITY GA. TIME START 1815
 SAMPLING LOCATION: CONDENSER EXHAUST TIME FIN. 1845
 DATE: 2-26-87 TEAM LEADER: BR TECHNICIANS: _____
 BAROMETRIC PRESSURE, IN. HG. (P_{BAR}): 30.40 PUMP #: M-25 TANK



TYPICAL LABELLING FOR TUBE A (a): 1-A-a

CLOCK TIME HOURS	ROTAMETER READINGS			
	A- SAMPLES	B- SAMPLES	C- SAMPLES	D- SAMPLES
0	48	45		
5	49	45		
10	49	45		
15	49	45		
20	49	45		
25	48	45		
30				

30
(MIN) NET TIME, MINUTES

CALI- BRATION	AMB. TEMP. (t _a)	1		2		C		D	
		TIME SECONDS (SEC)	VOLUME MILS (V _C)	TIME SECONDS	VOLUME MILS	TIME SECONDS	VOLUME MILS	TIME SECONDS	VOLUME MILS
INITIAL	44	39		36	→				
FINAL	43	39		38	→				
AVERAGE	44	60	39	60	38				
VOLUME LITERS (V _a)			1		1		1		1
VOLUME STANDARD (V _{sc})			dsl		dsl		dsl		dsl

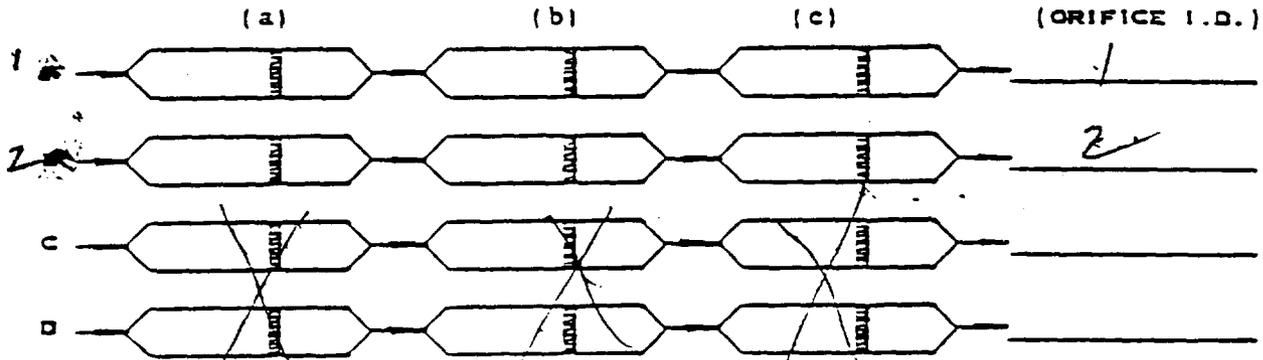
$$V_a = \text{MIN.} \cdot \frac{60}{1000} \cdot V_c / \text{SEC}$$

$$V_{sc} = 17.64 \cdot \frac{P_{bar}}{(t_a + 460)} \cdot V_a$$

ADSORBENT SAMPLING TRAIN FIELD DATA

COMPANY NAME <u>RESOURCE</u>	RUN <u>CE-2A</u>
ADDRESS <u>GARDEN CITY, CA.</u>	TIME START <u>2233</u>
SAMPLING LOCATION <u>CONDENSER EXHAUST</u>	TIME FIN. <u>2335</u>
DATE <u>2-26-87</u> TEAM LEADER <u>DR</u>	TECHNICIANS <u>TM</u>
BAROMETRIC PRESSURE, IN. HG. (P _{SAR}) <u>30.4</u>	PUMP # <u>M-25 TAJK</u>

2303



TYPICAL LABELLING FOR TUBE A (2): 1-A-2

CLOCK TIME HOURS	ROTAMETER READINGS			
	B- SAMPLES	B- SAMPLES	C- SAMPLES	D- SAMPLES
0	46	43		
5	46	43		
10	46	43		
15	46	43		
20	46	43		
25	46	43		
30	46	43		

30
NET TIME, MINUTES
(MIN)

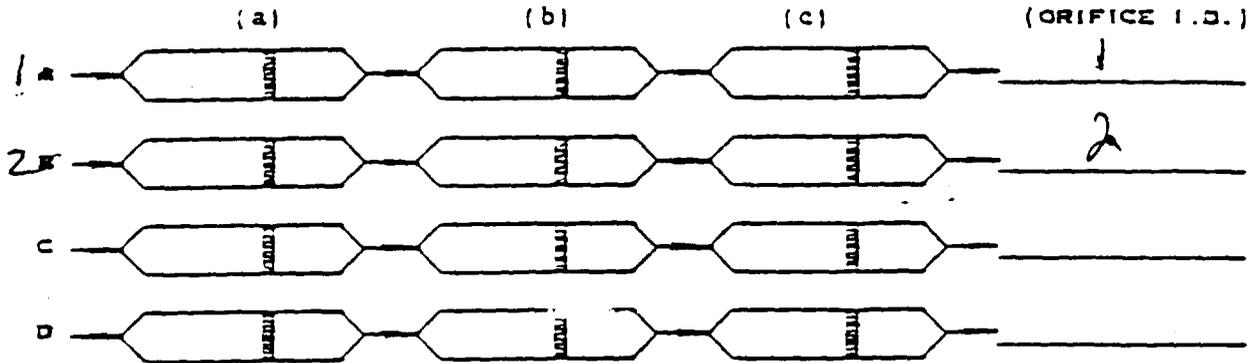
CALI- BRATION	AMB. TEMP. (T _a)	B		C		D	
		TIME SECONDS (SEC)	VOLUME MILS (V _c)	TIME SECONDS	VOLUME MILS	TIME SECONDS	VOLUME MILS
INITIAL	47		39		38		
FINAL	44		39		38		
AVERAGE	44	60	39	60	38		
VOLUME LITERS (V _a)			1		1		1
VOLUME STANDARD (V _{sc})			dsl		dsl		dsl

$$V_a = \text{MIN.} \cdot \frac{60}{1000} \cdot V_c / \text{SEC}$$

$$V_{sc} = 17.64 \cdot \frac{P_{SAT}}{(T_a + 460)} \cdot V_a$$

ADSORBENT SAMPLING TRAIN FIELD DATA

COMPANY NAME <u>RESOURCE CONSERVATION</u>			RUN <u>CE-2B</u>
ADDRESS <u>GARDEN CITY, GA.</u>			TIME START <u>2328</u>
SAMPLING LOCATION <u>CONDENSER EXHAUST</u>			TIME FIN. <u>2348</u>
DATE <u>2-26-87</u>	TEAM LEADER <u>DK</u>	TECHNICIANS <u>TM</u>	
BAROMETRIC PRESSURE, IN. HG. (P _{BAR}) <u>30.4</u>			PUMP # <u>M-25</u>



TYPICAL LABELLING FOR TUBE A (2): 1-A-2

CLOCK TIME	ROTAMETER READINGS				
	HOURS	1 - SAMPLES	2 - SAMPLES	C - SAMPLES	D - SAMPLES
2328	0	46	43		
	5	46	43		
	10	46	43		
	15	46	43		
	20	46	43		

20
(MIN) NET TIME, MINUTES

BYRON MODEL 90

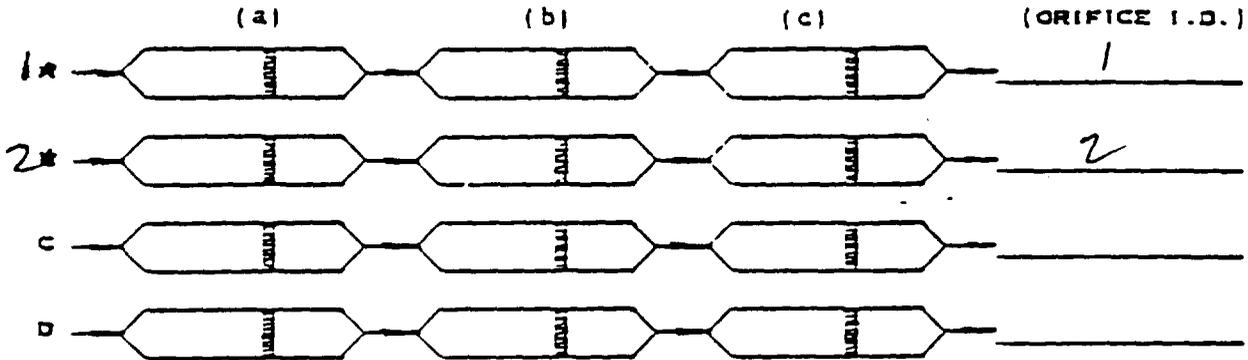
CALI- BRATION	AMB. TEMP. (T _a)	-1-		-2-		-C-		-D-	
		TIME SECONDS (SEC)	VOLUME MILS (V _c)	TIME SECONDS	VOLUME MILS	TIME SECONDS	VOLUME MILS	TIME SECONDS	VOLUME MILS
INITIAL	44		39		38				
FINAL	44		39		38				
AVERAGE	44	60	39	60	38				
VOLUME LITERS (V _a)			1		1		1		1
VOLUME STANDARD (V _{sc})			dsl		dsl		dsl		dsl

$$V_a = \text{MIN.} \cdot \frac{60}{1000} \cdot V_c / \text{SEC}$$

$$V_{sc} = 17.64 \cdot \frac{P_{bar}}{(T_a + 460)} \cdot V_a$$

ADSORBENT SAMPLING TRAIN FIELD DATA

COMPANY NAME <u>RESOURCE CONSERVATION</u>		RUN <u>CE-2C</u>
ADDRESS <u>GARDEN CITY, GA.</u>		TIME START <u>019</u>
SAMPLING LOCATION <u>CONDENSER EXHAUST</u>		TIME FIN. <u>049</u>
DATE <u>2-27-87</u>	TEAM LEADER <u>DC</u>	TECHNICIANS <u>TM</u>
BAROMETRIC PRESSURE, IN. HG. (P BAR) <u>30.4</u>		PUMP # <u>H-25 TANK</u>



TYPICAL LABELLING FOR TUBE A (2): 1-A-2

CLOCK TIME	ROTAMETER READINGS				
	HOURS	A- SAMPLES	B- SAMPLES	C- SAMPLES	D- SAMPLES
019	0	46	43		
	5	46	43		
	7	46	43		
	15	46	43		
	20	46	43		
	25	46	43		
	30	46	43		

30
(MIN)

NET TIME, MINUTES

BYRON - MODEL 90

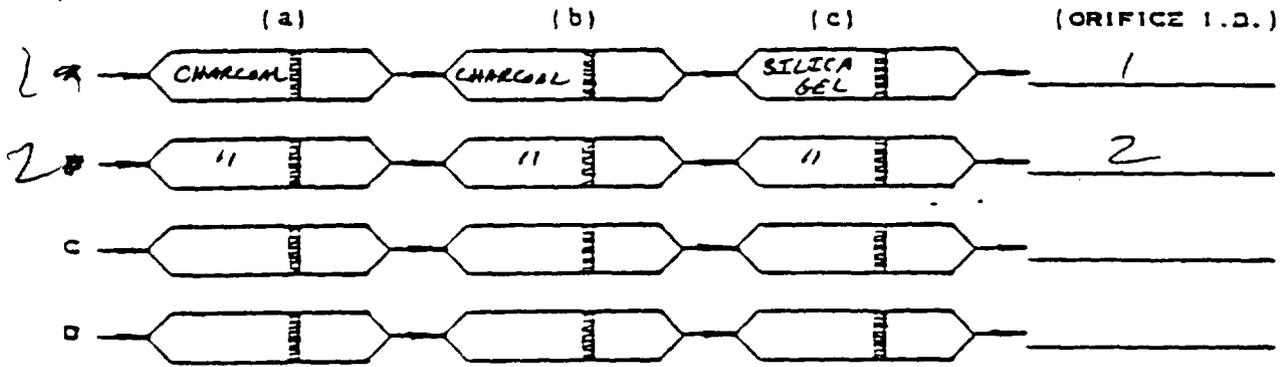
CALI- BRATION	AMB. TEMP. (Ca)	1-A		B-2		C-		D-	
		TIME SECONDS (SEC)	VOLUME MILS (Vc)	TIME SECONDS	VOLUME MILS	TIME SECONDS	VOLUME MILS	TIME SECONDS	VOLUME MILS
INITIAL	44		39		38				
FINAL	44		38		39				
AVERAGE	44	60	39	60	39				
VOLUME LITERS (Va)			1		1		1		1
VOLUME STANDARD (Vsc)			dsl		dsl		dsl		dsl

$$V_a = \text{MIN.} * \frac{60}{1000} * V_c / \text{SEC}$$

$$V_{sc} = 17.64 * \frac{P_{BAR}}{(Ca + 460)} * V_a$$

ADSORBENT SAMPLING TRAIN FIELD DATA

COMPANY NAME <u>RESOURCE CONSERVATION</u>	RUN <u>CE-3A</u>
ADDRESS <u>GARDEN CITY, GA.</u>	TIME START <u>03:15</u>
SAMPLING LOCATION <u>CONDENSOR EXHAUST</u>	TIME FIN. <u>03:45</u>
DATE <u>2/27/87</u> TEAM LEADER <u>BWJ</u>	TECHNICIANS _____
BAROMETRIC PRESSURE, IN. HG. (P _{SAR}) <u>30.4</u>	PUMP # _____



TYPICAL LABELLING FOR TUBE A (a): 1-A-a

CLOCK TIME HOURS	ROTAMETER READINGS			
	1 - SAMPLES	2 - SAMPLES	C - SAMPLES	D - SAMPLES
0315/0	45	42		
5	46	43		
10	46	43		
15	46	43		
20	46	43		
25	46	43		
0345/30				

30
(MIN) NET TIME, MINUTES

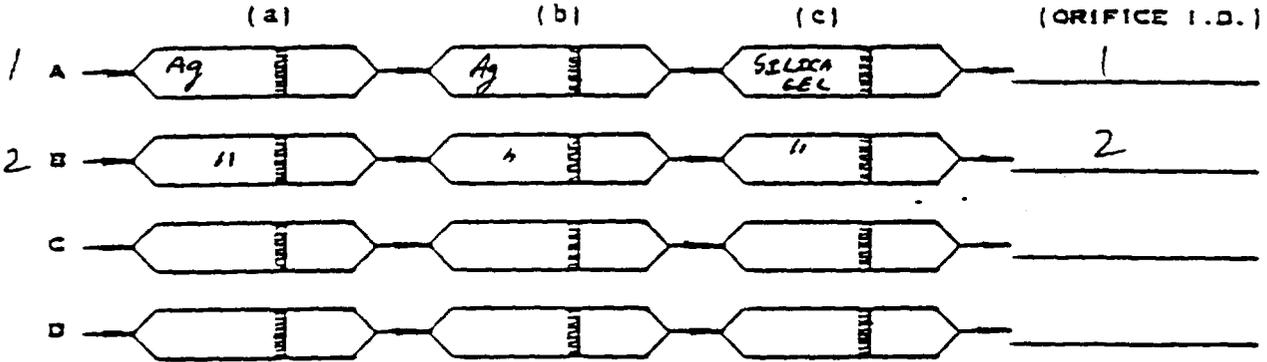
CALI- BRATION	AMB. TEMP. (T _a)	1		2		C		D	
		TIME SECONDS (SEC)	VOLUME MILS (V _c)	TIME SECONDS	VOLUME MILS	TIME SECONDS	VOLUME MILS	TIME SECONDS	VOLUME MILS
INITIAL	48	NA	39	NA	38				
FINAL	50	NA	39	NA					
AVERAGE	49	60	39	60	38				
VOLUME LITERS (V _a)			1		1		1		1
VOLUME STANDARD (V _{sc})			dsl		dsl		dsl		dsl

$$V_a = \text{MIN.} \cdot \frac{60}{1000} \cdot V_c / \text{SEC}$$

$$V_{sc} = 17.64 \cdot \frac{P_{SAR}}{(T_a + 460)} \cdot V_a$$

ADSORBENT SAMPLING TRAIN FIELD DATA

COMPANY NAME <u>Resource Conservation</u>	RUN <u>CE-3B</u>
ADDRESS <u>Hardee City, Ga.</u>	TIME START <u>0420</u>
SAMPLING LOCATION <u>Condenser Exhaust</u>	TIME FIN. <u>0450</u>
DATE <u>2/27/87</u> TEAM LEADER <u>BWH</u>	TECHNICIANS <u> </u>
BAROMETRIC PRESSURE, IN. HG. (P) <u>30.4</u>	PUMP # <u> </u>



TYPICAL LABELLING FOR TUBE A (2): 1-A-a

CLOCK TIME HOURS	ROTAMETER READINGS			
	1- SAMPLES	2- SAMPLES	C- SAMPLES	D- SAMPLES
0420 / 0	46	43		
5	46	43		
10	46	43		
15	46	43		
20	46	43		
25	46	43		
0450 / 30	46	43		

30
(MIN) NET TIME, MINUTES

CALI- BRATION	AMB. TEMP. (Ca)	1		2		C		D	
		TIME SECONDS (SEC)	VOLUME MILS (Vc)	TIME SECONDS	VOLUME MILS	TIME SECONDS	VOLUME MILS	TIME SECONDS	VOLUME MILS
INITIAL	50	NA	39	N/A	38				
FINAL	50		39		38				
AVERAGE	50	60	39	60	38				
VOLUME LITERS (Va)			1		1		1		1
VOLUME STANDARD (Vsc)			dsl		dsl		dsl		dsl

$$V_a = \text{MIN.} * \frac{60}{1000} * V_c / \text{SEC}$$

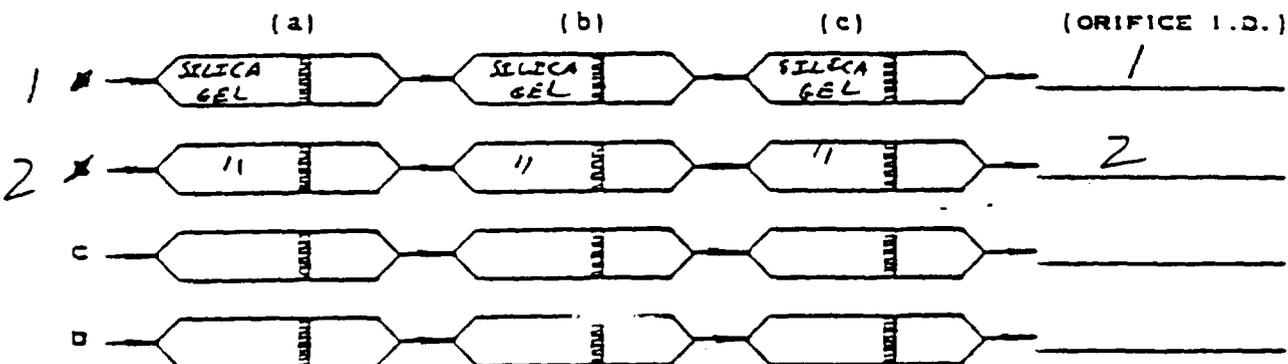
$$V_{sc} = 17.64 * \frac{P_{bar}}{(T_a + 460)} * V_a$$

2/28
F-1074
G-22

ENTROPY

ADSORBENT SAMPLING TRAIN FIELD DATA

COMPANY NAME <u>Resource Conservation</u>	RUN <u>CE-3C</u>
ADDRESS <u>Marden City, Mo</u>	TIME START <u>520</u>
SAMPLING LOCATION <u>Condenser Exhaust</u>	TIME FIN. <u>550</u>
DATE <u>2/27/87</u> TEAM LEADER <u>BWH</u>	TECHNICIANS _____
BAROMETRIC PRESSURE, IN. HG. (P _{BAR}) <u>30.4</u>	PUMP # _____



TYPICAL LABELLING FOR TUBE A (a): 1-A-2

CLOCK TIME HOURS	ROTAMETER READINGS			
	1- SAMPLES	2- SAMPLES	C- SAMPLES	D- SAMPLES
520/ 0	46	43		
5	46	43		
10	46	43		
15	46	43		
20	46	43		
25	46	43		
550/ 30	46	43		

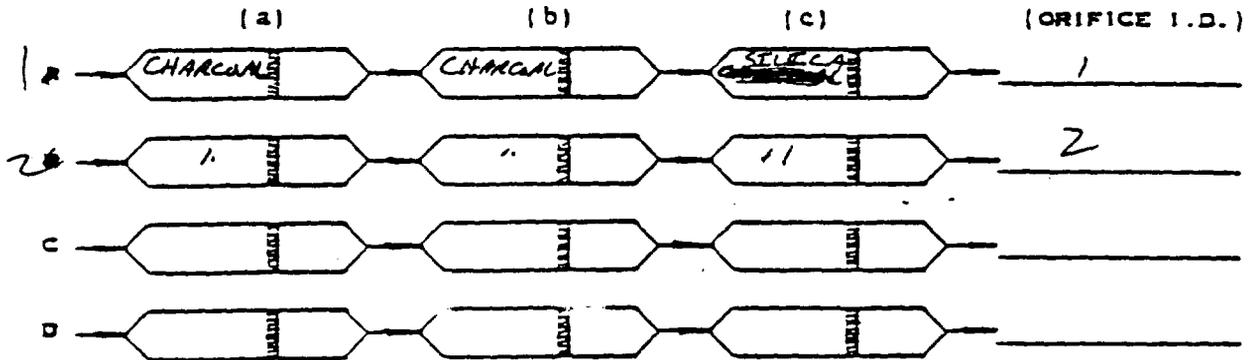
30
(MIN) NET TIME, MINUTES

CALI- BRATION	AMB. TEMP. (T _a)	1-*		2		C-		D-	
		TIME SECONDS (SEC)	VOLUME MILS (V _c)	TIME SECONDS	VOLUME MILS	TIME SECONDS	VOLUME MILS	TIME SECONDS	VOLUME MILS
INITIAL	50	NA	39	NA	38				
FINAL	50	NA	39	NA	38				
AVERAGE	50	60	39	60	38				
VOLUME LITERS (V _a)			1		1		1		1
VOLUME STANDARD (V _{sc})			dsl		dsl		dsl		dsl

$$V_a = \text{MIN.} \cdot \frac{60}{1000} \cdot V_c / \text{SEC} \qquad V_{sc} = 17.64 \cdot \frac{P_{bar}}{(T_a + 460)} \cdot V_a$$

ADSORBENT SAMPLING TRAIN FIELD DATA

COMPANY NAME <u>RESOURCES CONSERVATION CO</u>	RUN <u>CE-4A</u>
ADDRESS <u>GARDEN CITY, GA.</u>	TIME START <u>11:15</u>
SAMPLING LOCATION <u>CONDENSER EXHAUST</u>	TIME FIN. <u>11:45</u>
DATE <u>2/28/87</u> TEAM LEADER <u>BWB</u>	TECHNICIANS _____
BAROMETRIC PRESSURE, IN. HG. (P _{BAR}) <u>30.4</u>	PUMP # _____



TYPICAL LABELLING FOR TUBE A (a): 1-A-2

CLOCK TIME HOURS	ROTAMETER READINGS			
	1 - SAMPLES	2 - SAMPLES	C - SAMPLES	D - SAMPLES
11:15 / 0	46	43		
5	46	43		
10	46	43		
15	46	43		
20	46	43		
25	46	43		
30	46	43		

30
(MIN)

NET TIME, MINUTES

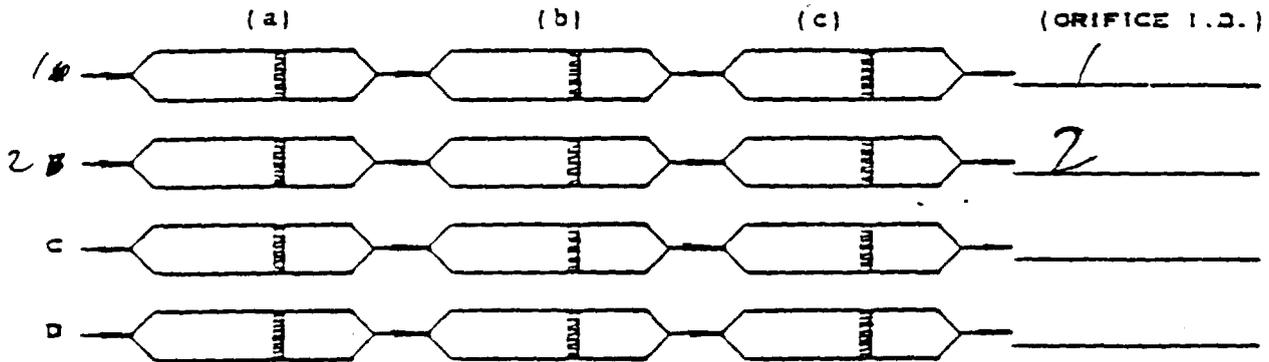
CALI- BRATION	AMB. TEMP. (T _a)	1 -		2 -		-C-		-D-	
		TIME SECONDS (SEC)	VOLUME MILS (V _c)	TIME SECONDS	VOLUME MILS	TIME SECONDS	VOLUME MILS	TIME SECONDS	VOLUME MILS
INITIAL	46	NA	39	NA	38				
FINAL	46	NA	39	NA	38				
AVERAGE	46	60	39	60	38				
VOLUME LITERS (V _a)			1		1		1		1
VOLUME STANDARD (V _{sc})			dsl		dsl		dsl		dsl

$$V_a = \text{MIN.} \cdot \frac{60}{1000} \cdot V_c / \text{SEC}$$

$$V_{sc} = 17.64 \cdot \frac{P_{bar}}{(T_a + 460)} \cdot V_a$$

ADSORBENT SAMPLING TRAIN FIELD DATA

COMPANY NAME <u>RESOURCES CONSERVATION</u>	RUN <u>CE-4B</u>
ADDRESS <u>GARDEN CITY GA.</u>	TIME START <u>12:00</u>
SAMPLING LOCATION <u>CONDENSER EXHAUST</u>	TIME FIN. <u>12:30</u>
DATE <u>2/28/87</u> TEAM LEADER <u>BWH</u>	TECHNICIANS _____
BAROMETRIC PRESSURE. IN. HG. (P BAR) <u>30.4</u>	PUMP # _____



TYPICAL LABELLING FOR TUBE A (a): 1-A-a

CLOCK TIME HOURS	ROTAMETER READINGS			
	A- SAMPLES	B- SAMPLES	C- SAMPLES	D- SAMPLES
0	46	43		
5	46	43		
10	46	43		
15	46	43		
20	46	43		
25	46	43		
30				

30
NET TIME, MINUTES
(MIN)

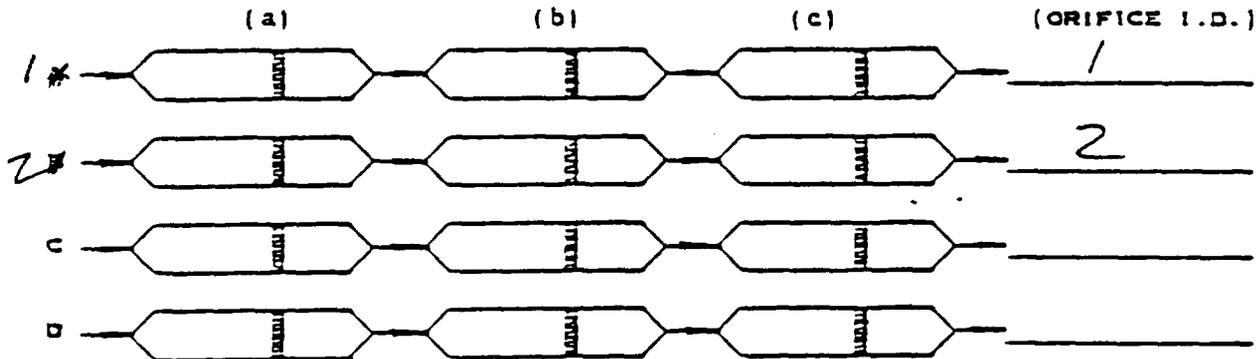
CALI- BRATION	AMB. TEMP. (t_a)	-A-		-B-		-C-		-D-	
		TIME SECONDS (SEC)	VOLUME MILS (V_c)	TIME SECONDS	VOLUME MILS	TIME SECONDS	VOLUME MILS	TIME SECONDS	VOLUME MILS
INITIAL	48	NA	39	NA	38				
FINAL	48	-	39	-	38				
AVERAGE	48	60	39	60	38				
VOLUME LITERS (V_a)			1		1		1		1
VOLUME STANDARD (V_{sc})			dsl		dsl		dsl		dsl

$$V_a = \text{MIN.} \cdot \frac{80}{1000} \cdot V_c / \text{SEC}$$

$$V_{sc} = 17.64 \cdot \frac{P_{bar}}{(t_a + 460)} \cdot V_a$$

ADSORBENT SAMPLING TRAIN FIELD DATA

COMPANY NAME <u>RESOURCES CONSERVATION</u>			RUN <u>CE-4C</u>	
ADDRESS <u>GARDEN CITY, GA.</u>			TIME START <u>1250</u>	
SAMPLING LOCATION <u>CONDENSER EXHAUST</u>			TIME FIN. <u>1320</u>	
DATE <u>2/28/87</u>	TEAM LEADER <u>RWBA</u>	TECHNICIANS <u> </u>		
BAROMETRIC PRESSURE, IN. HG. (P _{BAR}) <u>30.4</u>		PUMP # <u> </u>		



TYPICAL LABELLING FOR TUBE A (2): 1-A-2

CLOCK TIME HOURS	ROTAMETER READINGS			
	1-R. SAMPLES	2-R. SAMPLES	C. SAMPLES	D. SAMPLES
1250 / 0	46	43		
5	46	43		
10	46	43		
15	46	43		
20	46	43		
25	46	43		
1320 / 30				

30
(MIN)

NET TIME, MINUTES

CALI- BRATION	AMB. TEMP. (T _a)	1		2		C.		D.	
		TIME SECONDS (SEC)	VOLUME MILS (V _C)	TIME SECONDS	VOLUME MILS	TIME SECONDS	VOLUME MILS	TIME SECONDS	VOLUME MILS
INITIAL	46	NA	39	NA	38				
FINAL	46	"	39	"	38				
AVERAGE	46	60	39	60	38				
VOLUME LITERS (V _a)			1		1		1		1
VOLUME STANDARD (V _{sc})			dsl		dsl		dsl		dsl

$$V_a = \text{MIN.} \cdot \frac{80}{1000} \cdot V_c / \text{SEC}$$

$$V_{sc} = 17.64 \cdot \frac{P_{BAR}}{(T_a + 460)} \cdot V_a$$

APPENDIX B.2

B. FIELD DATA

2. Oil Polisher Outlet

EVACUATED TANK FIELD DATA

Column 96

Company Name RESOURCES CONSERVATION Run # OP-1
 Address Garden City, Mo Date 2/27/87
 Sampling Location Oil Polisher Outlet Start 1114
 Tank Number GT24 Tank Volume 7.071 liters Finish 1116.30
 SONIC ORIFICE ID - ~~191~~ cc Operator RWM

TANK PARAMETERS

Parameter	Barometric Pressure,		Tank Temp.,		Tank Pressure (mm Hg)		Leak Check (cm Hg change)	
	in. Hg	mmHg	F	C	Gauge	Absolute	Tank	System
Pretest	30.4	786	48	9	-756	162		
Post Test	30.4	772.2	48	9	-670	102.		
Final Pressure	30.4		48	9	+406			

at lab

29.12 66

$V_{sample} = 0.833 \text{ dsf}$

SAMPLE DATA

Clock Time	Gauge Pressure, Inches Hg	Flow Meter Setting, cc/min	TEMP
1114	0.05		
1115	.055		
1116	.105		
	.115		
1114	0.05		
1115	0.055		
1116	0.105		
1116.34	0.115		

APPENDIX C

ANALYTICAL DATA

ENTROPY

Report Number: 5448
Date Samples Received: 03/02/86
Date Samples Analyzed: 03/20/86
Analyst: Bruce Hawks
Report Date: 03/23/86

Subject: Analysis of a charcoal tubes for toluene, benzene, and xylenes.

Analytical Methodology: The entire contents of the charcoal tube was desorbed with 4.0 mL of carbon disulfide. Standards were prepared by spiking charcoal tubes with the analytes; the standard tubes were desorbed in the same manner as the sample tube thus inherently correcting for desorption efficiency. Analysis was performed on a Perkin-Elmer Model 8310 gas chromatograph with flame ionization detection. The chromatographic conditions were:

Analytical column 10% SP-1000 on 80/100 Supelcoport 20' x 1/8" S.S.
Column Temperature 100°C, hold 6 min, 20°C/min to 140°C, hold 4 min
Flow rate 40 mL/min, N₂
Injector Temperature 250°C
Detector Temperature 300°C

RESULTS: All results reported in micrograms per sample

Run ID	Benzene	Toluene	Xylenes
CE-1A-1a	1,260	760	1,070
CE-1A-1b	< 0.03	< 0.03	< 0.03
CE-2A-1a	1,260	668	971
CE-2A-1b	< 0.03	< 0.03	< 0.03
CE-3A-1a	1,330	673	1,020
CE-3A-1b	< 0.03	< 0.03	< 0.03
CE-4A-1a	1,220	610	862
CE-4A-1b	< 0.03	< 0.03	< 0.03
OP-1	106	111	389
OP-1	< 0.03	< 0.03	< 0.03


Bruce Hawks

Oxford Laboratories, Inc.

Analytical and Consulting Chemists

DATE RECEIVED 3-13-87
DATE REPORTED 3-24-87
87W6114

1316 South Fifth Street
Wilmington, N.C. 28401
(919) 763-9793

PAGE 1 OF 1

ENTROPY ENVIROMENTALIST INC
P.O. BOX 12291
RESEARCH TRIANGLE PK. , N.C. 27709-2291

P.O. # 5448A-3

ATTENTION: Bruce Hawks

SAMPLE DESCRIPTION: Bilayered .5N HNO3

1. OP-1 Reagent Upper Layer 1.5ml
2. OP-1 Reagent Lower Layer 46.5ml

RESULTS

	<u>1</u>	<u>2</u>
Mercury, ug/ml	X	<.01
Diethylamine, ug/ml	X	1500
Xylene, ug/ml	10,000	X
Toluene, ug/ml	1560	X
Benzene, ug/ml	<400	X

Bruce A. Babson
BRUCE A. BABSON , CHEMIST

Oxford Laboratories, Inc.

Analytical and Consulting Chemists

DATE RECEIVED 3-13-87
DATE REPORTED 3-12-87
87W6113

1316 South Fifth Street
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(919) 763-9793

PAGE 1 OF 2

ENTROPY ENVIROMENTALIST INC
P.O. BOX 12291
RESEARCH TRIANGLE PK. , N.C. 27709-2291

P.O. # 5448A-2

ATTENTION: Bruce Hawks

SAMPLE DESCRIPTION: 8 Silica Gel Tubes

1. CE-1B-1a
2. CE-1B-1b
3. CE-2B-1a
4. CE-2B-1b
5. CE-3B-1a
6. CE-3B-1b

RESULTS

	<u>1</u>	<u>2</u>	<u>3</u>	<u>4</u>	<u>5</u>	<u>6</u>
Triethylamine,mg/tube	78.3	<0.03	45.5	<0.03	157	<0.03

Oxford Laboratories, Inc.

Analytical and Consulting Chemists

DATE RECEIVED 3-13-87
DATE REPORTED 3-12-87
87W6113

1316 South Fifth Street
Wilmington, N.C. 28401
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PAGE 2 OF 2

ENTROPY ENVIROMENTALIST INC
P.O. BOX 12291
RESEARCH TRIANGLE PK. , N.C. 27709-2291

P.O. # 5448A-2

ATTENTION: Bruce Hawks

SAMPLE DESCRIPTION: 8 Silica Gel Tubes

- 7. CE-4B-1a
- 8. CE-4B-1b

RESULTS

	<u>7</u>	<u>8</u>
Triethylamine,mg/tube	152	<0.03

NOTE: See Discussion.


BRUCE A. BABSON , CHEMIST

Oxford Laboratories, Inc.

Analytical and Consulting Chemists

DATE RECEIVED 3-13-87
DATE REPORTED 3-12-87
87W6115

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Wilmington, N.C. 28401
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PAGE 1 OF 2

ENTROPY ENVIROMENTALIST INC
P.O. BOX 12291
RESEARCH TRIANGLE PK. , N.C. 27709-2291

P.O. # 5448A-1

ATTENTION: Bruce Hawks

SAMPLE DESCRIPTION: 8 tubes for Mercury

1. CE-1C-1a
2. CE-1C-1b
3. CE-2C-1a
4. CE-2C-1b
5. CE-3C-1a
6. CE-3C-1b

RESULTS

	<u>1</u>	<u>2</u>	<u>3</u>	<u>4</u>	<u>5</u>	<u>6</u>
Mercury as Hg, ug	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05

Oxford Laboratories, Inc.

Analytical and Consulting Chemists

DATE RECEIVED 3-13-87
DATE REPORTED 3-12-87
87W6115

1316 South Fifth Street
Wilmington, N.C. 28401
(919) 763-9793

PAGE 2 OF 2

ENTROPY ENVIROMENTALIST INC
P.O. BOX 12291
RESEARCH TRIANGLE PK. , N.C. 27709-2291

P.O. # 5448A-1

ATTENTION: Bruce Hawks

SAMPLE DESCRIPTION: 8 tubes for Mercury

7. CE-4C-1a
8. CE-4C-1b

RESULTS

Mercury as Hg, ug

<u>7</u>	<u>8</u>
<0.05	<0.05

Bruce A. Babson

BRUCE A. BABSON , CHEMIST

Oxford Laboratories, Inc.

Analytical and Consulting Chemists
1316 South Fifth Street
Wilmington, N.C. 28401
(919) 763-9793

March 19, 1987

Entropy Environmentalists Inc.
P.O. Box 12291
Research Triangle Park, N.C. 27709-2291

Attention: Bruce Hawks

Sample Description: 8 Silica Gel Tubes

DISCUSSION

"A" tubes were desorbed with 15ml of 1.0 NH₂SO₄. Two desorption efficiency trials were performed using this ratio of solvent to adsorbent. The data for recovery was as follows:

<u>Loading, mg</u>	<u>Recovery, %</u>
36.3	67.4
217.8	86.4

Since these loading rates bracketed the amounts found on the "A" tubes, an average % recovery (76.9) was calculated and data for the "A" was adjusted using this recovery.

Recovery from the "B" tubes was not as efficient, probably due to the fact that only 5ml of acid was used. Recovery at 2.9mg was 25.8%. Detection limits for the "B" tubes were adjusted accordingly.

Bruce A. Babson
Bruce A. Babson, Chemist

BAB/lo

ENTROPY

ENVIRONMENTALISTS INC.

POST OFFICE BOX 12291
RESEARCH TRIANGLE PARK
NORTH CAROLINA 27709-2291
919-781-3550

INTERLABORATORY SAMPLE TRANSFER CHAIN-OF-CUSTODY FORM

Please return this form along with the analytical results.

The samples referenced on ~~EEI~~ Purchase Order Number: 5448A-2 were
shipped via Pony Express on 3/9/87 by BH
to Oxford Labs

Samples received at Oxford Labs on 3-13-87
by Roger Oxford

Note any broken seals, leakage, spillage, and/or damage to the samples.

"OK"

ENTROPY

ENVIRONMENTALISTS INC.

POST OFFICE BOX 12291
RESEARCH TRIANGLE PARK
NORTH CAROLINA 27709-2291
919-781-3550

INTERLABORATORY SAMPLE TRANSFER CHAIN-OF-CUSTODY FORM

Please return this form along with the analytical results.

The samples referenced on EMI Purchase Order Number: 5448A-7 were
shipped via Pony Express on 3/10/87 by BH
to Oxford Lab

Samples received at Oxford Labs on 3-13-87
by Roger Offord

Note any broken seals, leakage, spillage, and/or damage to the samples.

ENTROPY

ENVIRONMENTALISTS INC.

POST OFFICE BOX 12291
RESEARCH TRIANGLE PARK
NORTH CAROLINA 27709-2291
919-781-3550

INTERLABORATORY SAMPLE TRANSFER CHAIN-OF-CUSTODY FORM

Please return this form along with the analytical results.

The samples referenced on ~~EEI~~ Purchase Order Number: 5448A-1 were
shipped via Pony Express on 3/9/87 by BH
to Oxford Lab

Samples received at Oxford Labs on 3-13-87
by Roger Oxford

Note any broken seals, leakage, spillage, and/or damage to the samples.

APPENDIX D

CALIBRATION DATA

ORIFICE CALIBRATIONS

Condenser Exhaust

By: BDR on 3-5-87

Orifice # 1
 Box N-5 Y = 1.0237 @ Dh<=0.5
 MolWt, wet = 28.68

Orifice Delta P	Meter Delta H	Volume acf	Time min	Meter Tmp-F	Orif Tmp-F	Orif Flow acfm	Bar P. In. Hg	Coeffnt Km
0.05	0.07	0.664	5	75	69	0.1344	30.05	0.763
0.10	0.13	0.982	5	78	69	0.1977	30.05	0.791
0.15	0.18	1.19	5	80	70	0.2391	30.05	0.780
0.20	0.25	1.359	5	82	70	0.2721	30.05	0.768
0.25	0.32	1.518	5	84	70	0.3028	30.05	0.763
0.30	0.37	1.655	5	86	70	0.3290	30.05	0.755
0.40	0.48	1.894	5	79	69	0.3807	30.04	0.761
0.50	0.6	2.124	5	80	69	0.4261	30.04	0.762
0.60	0.72	2.324	5	82	69	0.4645	30.04	0.757
Average								0.7666

BAROMETRIC PRESSURE (MM HG) <u>758.0</u> P_B AMBIENT TEMPERATURE ($^{\circ}$ C) <u>10</u> (T_A)	METER IDENTIFICATION <u>Leather Case</u> CALIBRATION DATE <u>2/26/87</u> CALIBRATION TIME <u>1109</u>
--- BUBBLE METER DATA --- RUN TIME (MINUTES) <u>0.96</u> (θ) MEASURED VOLUME. (LITERS) <u>0.10</u> (V_B)	FLOW RATE SETTING (MLPM) <u>100.0</u> METERED VOLUME. V_M (DRY STD. LITERS) <u>0.10</u> V_M
VOLUME STD., $V_{SB} = (.386)(P_B/(T_A + 273))(V_B) = 1.034$ FLOW RATE, STD = $0.1034 V_{SB} / (0.96 \theta) = 10.77$	CALIBRATION FACTOR (γ) $\gamma = 1.034 V_{SB} / (0.10 V_M) = 1.034$

BAROMETRIC PRESSURE (MM HG) <u>758.0</u> P_B AMBIENT TEMPERATURE ($^{\circ}$ C) <u>10</u> (T_A)	METER IDENTIFICATION <u>Leather Case</u> CALIBRATION DATE <u>2/26/87</u> CALIBRATION TIME <u>1109</u>
--- BUBBLE METER DATA --- RUN TIME (MINUTES) <u>0.95</u> (θ) MEASURED VOLUME. (LITERS) <u>0.10</u> (V_B)	FLOW RATE SETTING (MLPM) <u>100</u> METERED VOLUME. V_M (DRY STD. LITERS) <u>0.10</u> V_M
VOLUME STD., $V_{SB} = (.386)(P_B/(T_A + 273))(V_B) = 0.1034$ FLOW RATE, STD = $0.1034 V_{SB} / (0.95 \theta) = 0.1088$	CALIBRATION FACTOR (γ) $\gamma = 0.1034 V_{SB} / (0.10 V_M) = 1.034$

BAROMETRIC PRESSURE (MM HG) <u>758.0</u> P_B AMBIENT TEMPERATURE ($^{\circ}$ C) <u>10</u> (T_A)	METER IDENTIFICATION <u>Leather Case</u> CALIBRATION DATE <u>2/26/87</u> CALIBRATION TIME <u>1109</u>
--- BUBBLE METER DATA --- RUN TIME (MINUTES) <u>0.95</u> (θ) MEASURED VOLUME. (LITERS) <u>0.10</u> (V_B)	FLOW RATE SETTING (MLPM) <u>100</u> METERED VOLUME. V_M (DRY STD. LITERS) <u>0.10</u> V_M
VOLUME STD., $V_{SB} = (.386)(P_B/(T_A + 273))(V_B) = 0.1034$ FLOW RATE, STD = $0.1034 V_{SB} / (0.95 \theta) = 0.1088$	CALIBRATION FACTOR (γ) $\gamma = 0.1034 V_{SB} / (0.10 V_M) = 1.034$

APPENDIX E

SAMPLING AND ANALYTICAL PROCEDURES

ALIPHATIC AMINES IN AIR

Physical and Chemical Analysis Branch

Analytical Method

Analyte: Aliphatic Amines	Method No.: P&CAM 221
Matrix: Air	Range: 1 to 2400 mg/m ³ in a 10-liter sample of air
Procedure: Adsorption on silica gel; elution with acid; GC analysis	Precision (CV _T): 0.03 for methylamine at 300 mg/m ³
Date Issued: 1/29/76	Classification: E (Proposed)
Date Revised:	

1. Principle of the Method

- 1.1 A known volume of air is drawn through a tube containing silica gel to trap the aliphatic amines. The silica gel is transferred to glass-stoppered tubes and treated with sulfuric acid. A portion of the resulting acid solution is made alkaline with an excess of sodium hydroxide and an aliquot of the alkaline solution is analyzed by gas chromatography with a flame ionization detector.
- 1.2 The method may be used to determine a single aliphatic amine or to determine two or more amines in a single sample. The method has been applied to the following individual compounds:

Methylamine
Ethylamine
Dimethylamine
Isopropylamine
Butylamine
Diethylamine
Diisopropylamine
Triethylamine
Cyclohexylamine

2. Range and Sensitivity

- 2.1 The limit of detection of the method is 0.01 mg of amine (0.02 mg of methylamine) per sample when the analyte is desorbed from 150 mg of silica gel with 2 ml of sulfuric acid and a 3- μ l aliquot of the alkaline mixture is analyzed. This limit corresponds approximately to 1 mg/m³ (2 mg/m³ for methylamine) in a 10-liter sample of air.

4.6 A coefficient of variation of 0.03 has been determined for the analysis of 10 samples of 1.2 mg of methylamine collected from 4 liters of air (300 mg/m^3) with a personal sampling pump. The recovery was 99%.

5. Advantages and Disadvantages

5.1 The sampling method uses a small, portable device involving no liquids. The sorbent tube can be used for at least 8 hr to measure an average workday concentration, or for only 15 min to measure an excursion concentration. Desorption of the sample and preparation for analysis are simple. The analysis is accomplished by a rapid instrumental method.

5.2 Most analytical interferences can be eliminated by altering the GC conditions.

5.3 Several aliphatic amines can be collected and determined simultaneously. This is especially useful when the identity and composition of the amine vapors are not known.

5.4 The major disadvantage of the method is the necessity for measuring desorption efficiencies. When the desorption is less than complete, as for higher molecular weight amines, the effect of the amount of compound absorbed must also be determined.

6. Apparatus

6.1 Air Sampling Equipment

6.1.1 Sorbent tubes. The sorbent tubes consist of Pyrex glass tubes 125 mm long and 8 mm i.d., packed with three separate sections of 45/60-mesh activated silica gel. The weights of the three sections of silica gel are, in order, 600, 150, and 150 mg; these tubes are designed for sample flow in either direction. Plugs of 100-mesh stainless steel screen are used to contain the silica gel sections. These plugs of negligible pressure drop are prepared from 7-mm discs of screen held in place by Teflon rings. Pieces of Pyrex tubing 12 mm long and 7 mm in o.d. are located between the sorbent sections to inhibit migration of the amines. The ends of the tubes are flamesealed after packing to prevent contamination before use for sampling. The pressure drop across the tubes does not exceed 6 cm of water at a flow rate of 200 ml/min. The silica gel should be the equivalent of Silica Gel D-08, Chromatographic Grade, Activated and Fines Free, 45/60-mesh, as supplied by Applied Science Laboratories, Inc., State College, Pa. Polyethylene caps should be provided to seal the tubes after sampling has been completed.

8.2 Collection and Shipping of Samples

- 8.2.1 Immediately before beginning the collection of a sample, break each end of the sorbent tube so as to provide openings at least half the inside diameter of the tube.
- 8.2.2 Choose the direction desired for sample flow, mark the inlet end of the tube with a permanent marker, and attach the other end to the sampling pump. For low concentrations of amines, low humidity conditions, or short sampling periods, pump the sample air through the 150-mg sorbent section first; for high concentrations, high humidity, or long sampling periods, pump the sample air through the 600-mg sorbent section first. Sample air must not pass through any hose or tubing before entering the sorbent tube.
- 8.2.3 With the sorbent tube in a vertical position, sample the air at 200 mL/min for the desired period of time (0.25 to 8 hr). The flow rate and sampling time, or the volume, must be measured as accurately as possible. The temperature and pressure of the air being sampled should be measured and recorded.
- 8.2.4 Immediately after sampling is completed, cap the sorbent tubes with the polyethylene caps provided. Rubber caps must not be used.
- 8.2.5 One tube should be handled in the same manner as the sample tubes (break, seal, and ship) except that no air is pumped through it. Label this tube as a blank.
- 8.2.6 Pack the tubes tightly before shipping to minimize breakage in transit.
- 8.2.7 After the sample is collected the tubes should not be subjected to extremes of high temperature or low pressure. Refrigeration will reduce sample loss.

8.3 Analysis of Samples

- 8.3.1 **Preparation of Samples.** Remove and discard the stainless steel plugs and glass spacers and transfer each section of silica gel to a separate glass-stoppered test tube or flask. Label the samples and analyze each section separately.
- 8.3.2 **Desorption.** Desorb the amines from the silica gel by adding 2 mL of 1.0 *N* sulfuric acid to the 150-mg sections and 8 mL to the 600-mg section. Shake the sample mixtures occasionally over a period of 1 hr. Tests have indicated that desorption reaches a maximum in an hour.

8.4.1 **Importance of Determination.** Desorption efficiency for a particular compound can vary from one lot of silica gel to another, and also from one laboratory to another. Therefore, it is necessary to determine at least once the desorption efficiency for each amine with each lot of silica gel used. Among the aliphatic amines listed in Section 1.2 desorption efficiency decreases with increasing molecular weight. Desorption efficiency increases with increasing ratio of sulfuric acid to silica gel; however, an increase in this ratio results in a reduction in analytical sensitivity. A compromise ratio of 75 mg of silica gel per milliliter of sulfuric acid is recommended. Desorption efficiency may also vary with the amount of amine present on the silica gel. Measurements of efficiency should, therefore, be made for at least two amounts within the normal range of sample size.

8.4.2 **Procedure for Determination of Desorption Efficiency.** Place 150 mg of silica gel in a 2-ml glass-stoppered tube. The silica gel must be from the same lot as that used in collecting the sample; it can be obtained from unused sorbent tubes. With a microliter syringe, inject a known amount of the amine, either pure or in water solution, directly onto the silica gel. Close the tube with the glass stopper and allow it to stand at least overnight to insure complete adsorption of the amine. Prepare at least three tubes for each of at least two different amounts of amine. These tubes are referred to as samples. Prepare a blank in the same manner, omitting the amine. Analyze the samples and blank as described in Section 8.3. Also analyze three standards prepared by adding identical amounts of the amine to 2.0 ml of 0.1 N sulfuric acid. Determine the concentrations of the amine in the blank, samples, and standards using calibration curves prepared as described in Section 9. The desorption efficiency is calculated by dividing the concentration of amine found in the sample by the concentration obtained for the corresponding standard.

9. Standards and Calibration

9.1 For accuracy in the preparation of standards, it is recommended that an initial standard be prepared in a relatively large volume and at a high concentration. Aliquots of this standard can then be diluted to prepare other standards. Prepare the initial standard by pipetting an appropriate volume of amine into a 100-ml volumetric flask and adding distilled water to the mark. Check the concentration of this solution by titrating with standard sulfuric acid. Make the first dilution by adding an equal volume of 2.0 N sulfuric acid to an aliquot of the initial standard. Make additional dilutions with 1.0 N sulfuric acid so that the final concentration of sulfuric acid will be the same in standards and samples.

10.3 The concentration may also be expressed in terms of parts per million (ppm) by volume:

$$\text{ppm} = \text{mg/m}^3 \times \frac{24.45}{\text{M.W.}} \times \frac{760}{\text{P}} \times \frac{\text{T}+273}{298}$$

where: 24.45 = molar volume (ℓ/mole) at 25° C and 760 mmHg.

M.W. = molecular weight.

P = pressure (mmHg) of air sampled.

T = temperature (°C) of air sampled.

11. References

- 11.1 Campbell, Evan E., G.O. Wood, and R.G. Anderson, "Development of Air Sampling Techniques," Los Alamos Scientific Laboratory, Progress Reports LA-5634-PR (June 1974), LA-5973-PR (July 1975), and LA-6057-PR (September 1975).
- 11.2 Andre, C.E., and A.R. Mosier, "Precolumn Inlet System for the Gas Chromatographic Analysis of Trace Quantities of Short-Chain Aliphatic Amines," Anal. Chem., 45, 1971 (1973).
- 11.3 Wood, G. O., and R. G. Anderson, "Development of Air Monitoring Techniques Using Solid Sorbents," Los Alamos Scientific Laboratory, Progress Reports LA-6216-PR (February 1976) and LA-6513-PR (September 1976).

FORMULA: Table 1

HYDROCARBONS, AROMATIC

M.W.: Table 1

METHOD: 1501
ISSUED: 2/15/84

OSHA, NIOSH, ACGIH: Table 2

PROPERTIES: Table 1

COMPOUNDS:	benzene	cumene	α -methylstyrene	styrene	vinyltoluene
(Synonyms in Table 1)	<u>p-tert</u> -butyltoluene	ethylbenzene	naphthalene	toluene	xylene

SAMPLING	MEASUREMENT
SAMPLER: SOLID SORBENT TUBE (coconut shell charcoal, 100 mg/50 mg)	! TECHNIQUE: GAS CHROMATOGRAPHY, FID ! ! ANALYTES: hydrocarbons listed above !
FLOW RATE, VOLUME: Table 3	! DESORPTION: 1 mL CS ₂ ; stand 30 min !
SHIPMENT: no special precautions	! INJECTION VOLUME: 5 μ L !
SAMPLE STABILITY: not determined	! TEMPERATURE-INJECTION: 225 °C ! -DETECTOR: 225 °C !
BLANKS: 2 to 10 field blanks per set	! -COLUMN: see step 11 !
BULK SAMPLE: desirable, 1 to 10 mL; ship in separate containers from samples	! CARRIER GAS: N ₂ or He, 25 mL/min ! ! COLUMN: glass, 3.0 m x 2 mm, 10% OV-275 on 100/120 mesh Chromosorb W-AW or equivalent !
ACCURACY	!
RANGE STUDIED, BIAS and OVERALL PRECISION (s _p): Table 3	! CALIBRATION: analytes in CS ₂ ! ! RANGE AND PRECISION (s _p): Table 4 ! ! ESTIMATED LOD: 0.001 to 0.01 mg per sample with capillary column [1] !

APPLICABILITY: This method is for peak, ceiling and TWA determinations of aromatic hydrocarbons. It may be used for simultaneous measurements, though there is the possibility that interactions between analytes may reduce the breakthrough volumes and change desorption efficiencies.

INTERFERENCES: Use of the recommended column will prevent interference by alkanes (\leq C₁₀). Under conditions of high humidity, the breakthrough volumes may be reduced by as much as 50%. Other volatile organic solvents, e.g., alcohols, ketones, ethers and halogenated hydrocarbons, are possible interferences. If interference is suspected, use a less polar column or change column temperature.

OTHER METHODS: This method is based on and supercedes Methods P&CAM 127, benzene, styrene, toluene and xylene [2]; S311, benzene [4]; S22, p-tert-butyltoluene [3]; S23, cumene [3]; S29, ethylbenzene [3]; S26, α -methylstyrene [3]; S292, naphthalene [4]; S30, styrene [3]; S343, toluene [4]; S25, vinyltoluene [3]; S318, xylene [4].

9. Determine desorption efficiency (DE) at least once for each batch of charcoal used for sampling in the calibration range (step 8). Prepare three tubes at each of five levels plus three media blanks.
 - a. Remove and discard back sorbent section of a media blank sampler.
 - b. Inject a known amount of analyte (calibration stock solution for naphthalene) directly onto front sorbent section with a microliter syringe.
 - c. Cap the tube. Allow to stand overnight.
 - d. Desorb (steps 5 through 7) and analyze together with working standards (steps 11, 12 and 13).
 - e. Prepare a graph of DE vs. mg analyte recovered.
10. Analyze three quality control blind spikes and three analyst spikes to insure that the calibration graph and DE graph are in control.

MEASUREMENT:

11. Set gas chromatograph according to manufacturer's recommendations and to conditions given on page 1501-1. Select appropriate column temperature:

Substance ^a	Approximate Retention Time (min), at Indicated Column Temperature			
	50 °C	100 °C	150 °C	Programmed ^b
benzene	2.5			2.5
toluene	4.3	1.1		4.2
xylene (<i>para</i>)	7.0	1.4		5.2
ethylbenzene	7.0	1.4		5.5
xylene (<i>meta</i>)	7.2	1.5		5.6
cumene	8.3	1.6		6.0
xylene (<i>ortho</i>)	10	1.9		6.5
styrene	16	2.6		7.6
α -methylstyrene		3.2	1.0	8.1
vinyltoluene (<i>meta</i>)		3.8	1.2	8.5
naphthalene		25	4.3	12

^aData not available for *p-tert*-butyltoluene and *p*-vinyltoluene.

^bTemperature program: 50 °C for 3 min, then 15 °C/min to 200 °C.

NOTE: Alternatively, column and temperature may be taken from Table 4.

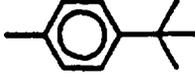
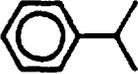
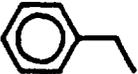
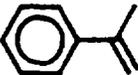
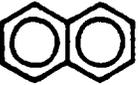
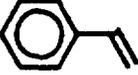
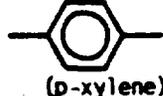
12. Inject sample aliquot manually using solvent flush technique or with autosampler.
NOTE: If peak area is above the linear range of the working standards, dilute with eluent, reanalyze and apply the appropriate dilution factor in calculations.
13. Measure peak area.

CALCULATIONS:

14. Determine the mass, mg (corrected for DE) of analyte found in the sample front (W_f) and back (W_b) sorbent sections, and in the average media blank front (B_f) and back (B_b) sorbent sections.

NOTE: If $W_b > W_f/10$, report breakthrough and possible sample loss.

Table 1. Synonyms, formula, molecular weight, properties [5].

Name/Synonyms	Structure	Empirical Formula	Molec- ular Weight	Boiling Point (°C)	Vapor Pressure @ 25 °C		Density @ 20 °C (g/mL)
					(mm Hg)	(kPa)	
benzene CAS #71-43-2		C ₆ H ₆	78.11	80.1	95.2	12.7	0.879
p-tert-butyltoluene CAS #98-51-1 1-tert-butyl-4-methylbenzene		C ₁₁ H ₁₆	148.25	192.8	0.7	0.09	0.861
cumene CAS #98-82-8 isopropylbenzene		C ₉ H ₁₂	120.20	152.4	4.7	0.62	0.862
ethylbenzene CAS #100-41-4		C ₈ H ₁₀	106.17	136.2	9.6	1.28	0.867
α-methylstyrene CAS #98-83-9 isopropenylbenzene (1-methylethenyl)-benzene		C ₉ H ₁₀	118.18	165.4	2.5	0.33	0.911
naphthalene CAS #91-20-3		C ₁₀ H ₈	128.18	80.2 ^a	0.2	0.03	1.025
styrene CAS #100-42-5 vinylbenzene		C ₈ H ₈	104.15	145.2	6.1	0.81	0.906
toluene CAS #108-88-3 methylbenzene		C ₇ H ₈	92.14	110.6	28.4	3.79	0.867
vinyltoluene ^b CAS #25013-15-4 methylstyrene methylvinylbenzene		C ₉ H ₁₀	118.18	167.7 171.6 172.8 169.8	1.6 1.9 1.8 1.8	0.22 0.26 0.24 0.24	0.898 0.911 0.911 0.904
xylene ^c CAS #1330-20-7 dimethylbenzene		C ₈ H ₁₀	106.17	144.4 139.1 138.4	6.7 8.4 8.8	0.89 1.12 1.19	0.880 0.864 0.861

^aMelting point.^bCommercial mixture of meta and para isomers.^cMixture of isomers.

Table 4. Measurement range, precision and conditions^a [3,4,12].

Substance	Desorption Volume (mL)	Measurement		Carrier Flow (mL/min)	Column Parameters ^b		
		Range (mg)	Precision (s _r)		t (°C)	Length (m)	Packing ^c
benzene	1.0	0.09- 0.35	0.036	50	115	0.9	A
p-tert-butyltoluene	0.5	0.27- 1.09	0.021 ^d	50	115	3.0	B
cumene	0.5	0.86- 3.46	0.010	50	99	3.0	B
ethylbenzene	0.5	2.17- 8.67	0.010	50	85	3.0	B
α-methylstyrene	0.5	0.69- 3.57	0.011	50	115	3.0	B
naphthalene	1.0	4.96-19.7	0.019	30	125	3.0	C
styrene	0.5	2.17- 8.49	0.013 ^d	50	109	3.0	B
toluene	1.0	1.13- 4.51	0.011	50	155	0.9	D
vinyltoluene	0.5	2.41- 9.64	0.008	50	120	3.0	B
xylene	1.0	2.00-10.4	0.010	50	180	0.9	D

^aInjection volume, 5.0 μL; nitrogen carrier gas.

^bAll columns stainless steel, 3.2 mm outside diameter.

^cA, 50/80 mesh Porapak P; B, 10% FFAP on 80/100 mesh Chromosorb W AW-DMCS;

C, 10% OV-101 on 100/120 mesh Supelcoport; D, 50/80 mesh Porapak Q.

^dCorrected value, calculated from data in [12].

MEASUREMENT:

8. Set up the measurement system as described in the APPENDIX.
9. Purge the system before analyzing samples and standards by heating the first desorption section 60 sec, allowing to cool 60 sec and heating second section 30 sec.
10. Allow system to cool 1 min before analyzing samples.
11. Measure samples and standards by inserting the tube, thermally desorbing first section 60 sec, waiting 60 sec and desorbing second section for 30 sec.
NOTE: Handle the tube only with lint-free gloves or tissues to prevent contamination.
12. Allow the system to cool at least 30 sec before removing tube and inserting the next sample.
CAUTION: Tubes are hot when removed. Catch them in a dry beaker as they come out of the desorption unit.

CALCULATIONS:

13. Read the absorbance of the sample and calculate the mass of Hg on the samples, W (μg), and blanks from the calibration graph. Obtain an average value for the media blanks, B (μg).
14. Calculate the concentration, C (mg/m^3), of mercury in air volume sampled, V (L):

$$C = \frac{W - B}{V}, \text{ mg}/\text{m}^3.$$

EVALUATION OF METHOD:

Method S199 [2] was issued on July 8, 1977, and validated over the range 0.046 to 0.18 mg/m^3 at 760 mm Hg and 18.0 °C using a 3-L sample [6]. Overall precision, s_p , was 0.061 with average recovery of 100.7%. The mercury concentration was independently determined by theoretical calculations based on mercury vapor pressure data and flow rate. Breakthrough did not occur after sampling 3.5 hrs at 0.184 L/min (39 L) in a Hg concentration of 0.244 mg/m^3 and 85% relative humidity. Six samples stored at room temperature for seven days gave results not significantly different from samples that were analyzed immediately after exposure.

REFERENCES:

- [1] Criteria for a Recommended Standard...Occupational Exposure to Inorganic Mercury, U.S. Department of Health, Education, and Welfare, Publ. (NIOSH) 73-11024 (1973).
- [2] Backup Data Report No. S199, prepared under NIOSH Contract 210-76-0123, available as "Ten NIOSH Analytical Methods, Set 5," available as Order No. PB 287-499 from NTIS, Springfield, VA 22161 (1977).
- [3] NIOSH Manual of Analytical Methods, 2nd. ed., V. 4, S199, U.S. Department of Health, Education, and Welfare, Publ. (NIOSH) 78-175 (1978).
- [4] Ibid, V. 5, P&CAM 175, U.S. Department of Health, Education, and Welfare, Publ. (NIOSH) 79-141 (1979).
- [5] Trujillo, P. E. and E. E. Campbell. Development of a Multistage Air Sampler for Mercury, *Anal. Chem.*, 47, 1629-1634 (1975).
- [6] NIOSH Research Report-Development and Validation of Methods for Sampling and Analysis of Workplace Toxic Substances, U.S. Department of Health and Human Services, Publ. (NIOSH) 80-133 (1980).

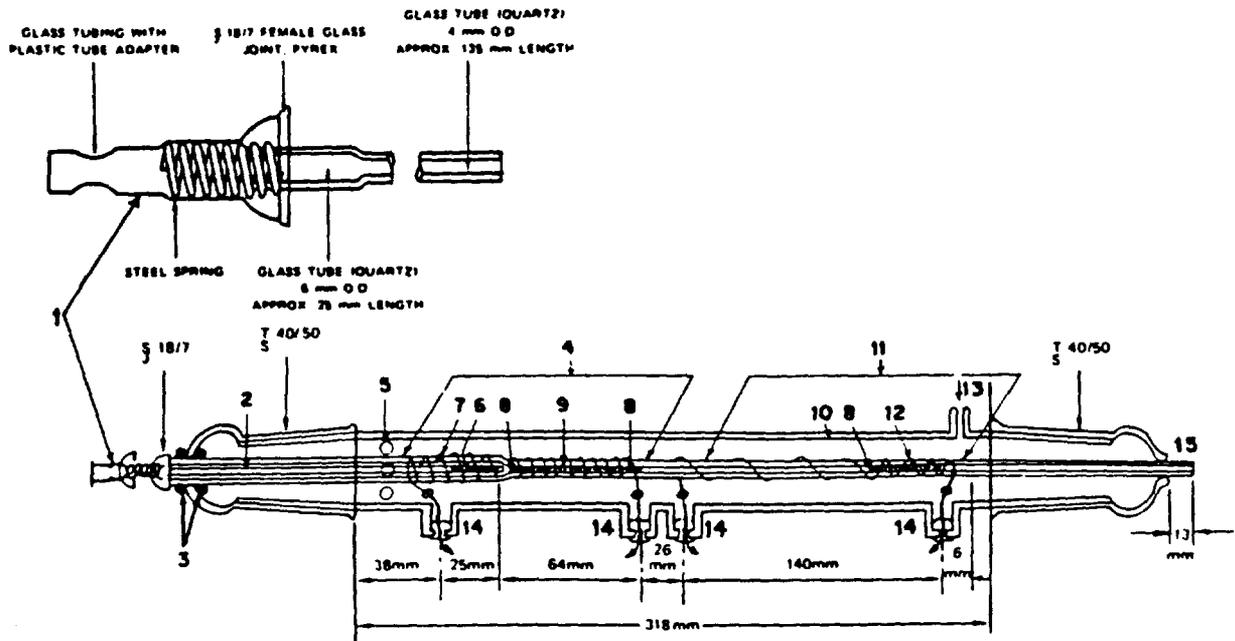
METHOD REVISED BY: R. DeLon Hull, NIOSH/DPSE; S199 originally validated under NIOSH Contract CDC-99-76-123.

- C. Carrier Air Supply. Room air is drawn by vacuum pump, attached to a ballast tank to prevent fluctuations in airflow.

Before entering the desorption unit, the air passes through a drying tube containing anhydrous $\text{Mg}(\text{ClO}_4)_2$ or other suitable drying agent. The dimensions of the drying tube and subsequent filters are not critical but should not impede airflow. Change the drying agent periodically, depending on the humidity of the air. The dried air passes through a filter containing activated charcoal and 30/60 mesh silvered Chromosorb P or Mersorb charcoal to remove organic vapors and metallic mercury before entering the thermal desorption unit.

Carrier air passes through, in order, the thermal desorption unit, the detection system, a second filter containing Mersorb charcoal which collects the mercury, a 0 to 5 L/min flowmeter, critical orifice, and into the ballast tank of the vacuum pump.

- D. Cooling air. Supplied by an air compressor at a flow rate of 20 L/min. The air enters the desorption unit near the second desorption section.
- E. Detection system. Mercury from the outlet of the thermal desorption unit enters the optical cell of a flameless atomic absorption spectrophotometer through a glass tube maintained at 70 °C using a heating tape. Power for the heating tape is supplied by a 120 V ac, 8 A variable transformer.



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| 1. LOADING MECHANISM | 8. QUARTZ GLASS WOOL PLUGS |
| 2. QUARTZ GLASS PLUNGER TUBE | 9. CUPRIC OXIDE (CuO) |
| 3. RUBBER 'O' RINGS | 10. COOLING JACKET |
| 4. SAMPLE DESORPTION SECTION | 11. SECOND DESORPTION SECTION |
| 5. COOLING AIR VENT HOLES | 12. GOLD SECTION |
| 6. SAMPLING TUBE | 13. COOLING AIR INTAKE |
| 7. NICHROME WIRE COILS | 14. WIRE HOLES AND SOLDERLESS CONNECTORS |
| | 15. OUTLET |

Figure 6000-2 THERMAL DESORPTION UNIT